REMOVAL OF CYANIDE USING PHOTOCATALYSIS-MEMBRANE HYBRID SYSTEM

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A project report submitted in partial fulfilment of the
requirements for the award of the degree of
Bachelor (Hons.) of Chemical Engineering

Faculty of Engineering and Science
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DECLARATION

I hereby declare that this project report is based on my original work except for citations and quotations which have been duly acknowledged. I also declare that it has not been previously and concurrently submitted for any other degree or award at UTAR or other institutions.

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APPROVAL FOR SUBMISSION

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Date : _________________________

Specially dedicated to
my beloved grandmother, mother and father

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ABSTRACT

This study was focused on removing cyanide compound from water using a photocatalysis-membrane hybrid system. Photocatalyst of TiO_2 (P25 Degussa) powder was used as pre-treatment of the system, whereas the hollow fiber membrane was used as the inline treatment and filtered out photocatalyst from treated water. The experiment setup used is relatively cheap, easy to set up and suitable for household and remote area. The effect of TiO_2 concentration, UV light power, and cyanide concentration on the removal efficiency of cyanide in water as well as the first-order rate constant were investigated. The removal efficiency was found to be increased with reaction time. The highest removal efficiency and rate constant were happen when 1.0 g/L of TiO_2 was used. This was followed by 0.15, 0.5 and 2.0 g/L of TiO_2. Moreover, the removal efficiency and rate constant also increased with the increase of light power used. Increasing initial concentration of cyanide had slightly increased the rate constant and removal efficiency. However 0.09 mg/L of cyanide was determined as the highest removal efficiency among all concentrations. The total recovery of photocatalyst by this experiment's module was in the range of 95.7 to 96.5%, depends on the initial TiO_2 concentration used.

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C	concentration of cyanide (mg/L) at time t
C_F	feed concentration of photocatalyst (mg/L)
C_o	initial concentration of cyanide (mg/L)
C_P	permeate concentration of photocatalyst (mg/L)
C_T	circumference of reactor tank (cm)
C_W	circumference of working volume (cm)
D_T	diameter of reactor tank (cm)
D_W	diameter of working volume (cm)
e^-	negatively charge electron
E_{bg}	energy gap band
E_{hg}	energy of photon
h^+	positively charge hole
H_T	tank height (m)
H_W	water level height (m)
k	first-order rate constant (h^{-1})
pH	potentiometric hydrogen ion concentration (dimensionless)
r	rate of photo-degradation of cyanide (mg/L h)
R	total recovery of photocatalyst (%)
t	reaction time (h)
V_T	tank volume (L)
V_W	working volume (L)
η	removal efficiency (%)
λ_{max}	maximum wavelength (nm)

CN^-	cyanide ion
CPCB	Central Pollution Control Board
EDTA	ethylenediaminetetraacetate
HCl	hydrochloric acid
HCN	hydrogen cyanide
LPM	litre per minute
MAC	maximum allowable concentration
MF	microfiltration
MINAS	Minimal National Standard Limit, India
NaOH	sodium hydroxide
NF	nanofiltration
NO_X	nitrogen oxides
PP	polypropylene
PTFC	polytetrafluoroethylene
RO	reverse osmosis
TiO_2	titanium dioxide
TSS	total suspended solid
UF	ultrafiltration
US EPA	United States Environmental Protection Agency
UV	ultraviolet
W	watt

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CHAPTER 1

INTRODUCTION

1.1 Background

Water supplies are becoming increasingly polluted with biological and chemical compounds, which are mainly caused by human activities such as industrial waste, agricultural waste, pesticides and so on. Polluted water is not safe to be consumed as it will lead to fatigue and sickness to human. The existing methods of wastewater treatment occasionally do not guarantee to produce effluent of safe and reusable quality. Especially in remote area, water treatment plant is not installed due to high installation and processing costs. Therefore, a low cost with advanced treatment technology needs to be further developed.

Cyanide is one of the water contaminants due to industry activities. In manufacturing, cyanide is used to make paper, textiles, and plastics. It is present in the chemicals used to develop photographs. Besides that, cyanide salts are used in metallurgy for electroplating, metal cleaning, and removing gold from its ore. Cyanide gas is used to exterminate pests and vermin in ships and buildings. The cyanide can enter the soil and then to the water body which causes water contaminated. Cyanide is acutely toxic to human. The exposure of cyanide will cause headache, drowsiness, vertigo, dermatitis, itching, weak and rapid pulse, nausea and vomiting. However, the over exposure of cyanide can cause brain damage, coma and even fatal (Hydrogen Cyanide, 1996).

The most common treatments of cyanide in water are using sulfur, iron salts, or chlorine. However, these methods are facing several challenges and limitations. High energy is required to convert cyanide to thiocyanate by using sulfur. Iron salts react with cyanide will form toxic ferrous complex and can cause ecological side effects. Moreover, chlorine itself is toxic because when it is generated, it produces nascent oxygen in the atomic state. Atomic oxygen is an aggressive oxidizing agent that can destroy living tissues (Gáspár, 2000).

Membrane technology has been already established as one of the reliable technology, but it has some limitations. Apart from the high power consumption, the fouling of the membrane reduces permeate flux and results in high operating cost (Shon et al., 2008). Beside membrane property and operating conditions, membrane fouling is mainly caused by the solute content in wastewater (Hu et al., 2004).

Heterogeneous photocatalytic oxidation processes is a green technology for water and wastewater treatment especially in removal of trace organics. By using photocatalysis, metal oxide catalysts used such as titanium oxide (TiO_2) generate hydroxyl radicals in the presence of ultraviolet (UV) light which can react with the pollutants in water to produce harmless species such as CO_2 and H_2O (Shon et al., 2008). The TiO_2 photocatalysis is commonly used because it is able to remove a wide range of pollutants.

In this study, an integrated photocatalysis-membrane hybrid system was used to remove cyanide in water. This system was chosen because it is relatively cheap, easy to set up and also able to reduce the fouling of the membrane. Photocatalytic reaction degrades and modifies the organic foulant in wastewater, and hence reduces the foulant on the membrane surface (Shon, 2006).

1.2 Objectives

The aim of this study was to remove cyanide in water using photocatalysis-membrane hybrid system. Some parameters under various conditions were investigated to study the removal efficiency, first-order rate constant and also to obtain the most suitable conditions for the module. Below are the objectives of this study:

(1) To determine the effect of TiO_2 concentration on the cyanide removal efficiency and the rate constant.
(2) To determine the effect of UV light power on the cyanide removal efficiency and the rate constant.
(3) To determine the effect of cyanide concentration on the removal efficiency and the rate constant.

1.3 Scopes of Study

A photocatalysis-membrane hybrid system was used in this study, where the hollow fibre membrane was chosen as the membrane of the system. In order to achieve the objectives of this study, the following scopes of work have been identified:

(1) Calibrating the cyanide concentration from a prepared standard solution before preparing the desired concentration of cyanide in water.
(2) Studying the removal efficiency and rate constant under different TiO_2 concentrations and choosing the most effective TiO_2 for further studies.
(3) Studying the removal efficiency and rate constant under various UV light powers and choosing the most effective light intensity for further studies.
(4) Investigating the removal efficiency and rate constant under different cyanide concentrations.
(5) Analysing the average of total suspended solid in water using the photocatalysis-hybrid membrane system.

1.4 Hypothesis

Based on the objectives in this study, the following hypotheses are proposed:

(1) The higher the concentration of TiO_2, the higher the removal efficiency.
(2) The higher the UV light power, the higher the removal efficiency.
(3) The higher the concentration of cyanide, the lower the removal efficiency.

CHAPTER 2

LITERATURE REVIEW

2.1 Membrane Technology

2.1.1 Description

Membrane technology has been already established as one of the reliable technology in water treatment. Membrane filtration is a pressure or vacuum driven separation process in which particulate matter larger than the pore of membrane will be rejected to pass through the barrier (Membrane Filtration Guidance Manual, 2005). The choice of membrane depends on the molecular size of the pollutants to be treated. There are four common classifications of membrane filtration, which are microfiltration (MF), ultrafiltration (UF), nonafiltration (NF) and reverse osmosis (RO). Figure 2.1 shows the process of membrane filtration, taken from Membrane Filtration Handbook by Jorgen Wagner (2001).

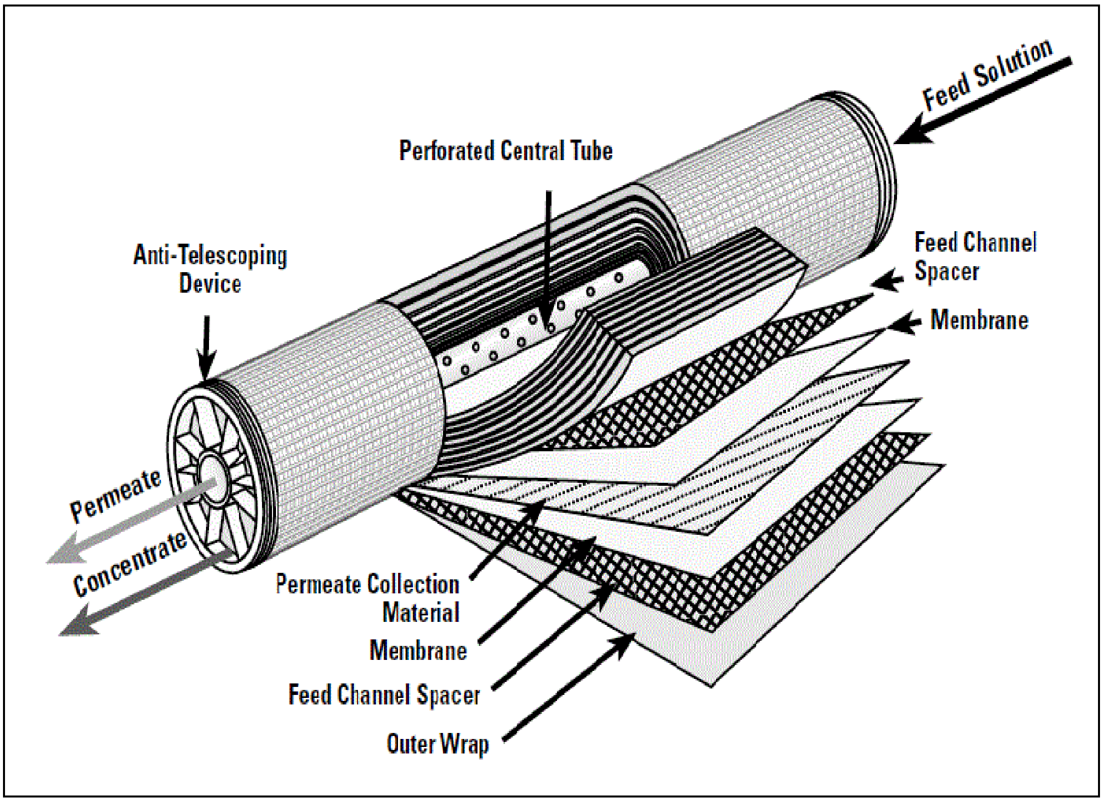


Figure 2.1: Process of Membrane Filtration

2.1.2 Advantages

Membrane filtration is a purely physical separation process, which does not involve a phase change or interphase mass transfer (Augugliaro et al., 2006). Besides, this technology offers an efficient way to improve water quality without destroying the fundamental sensory qualities of the water (Membrane Filtration, 2005). It removes microorganisms, such as bacteria, viruses and protozoa, which have a negative impact on water quality. Furthermore, membrane filtration is able to remove suspended solids without the use of chemicals. With less chemical consumption in water treatment, health risk related to chemical use is eliminated. The selection of membrane allows the selective type of materials being filtered out, as illustrated in Figure 2.2.

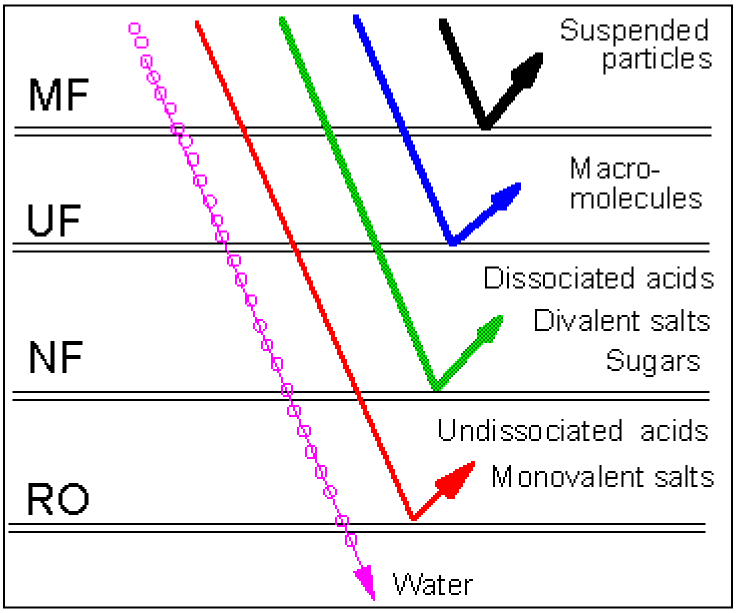


Figure 2.2: Types of Materials Filtered Out by Different Membranes

2.1.3 Disadvantages

There are some disadvantages of membrane filtration, especially microfiltration and ultrafiltration in water treatment. Apart from requiring high power consumption, membrane filtration also leads to foul on membrane, which significantly reduces the permeate flux in the filtration process. Membrane fouling is mainly caused by the solute content in wastewater (Shon et al. 2008).

Shon et al. (2008) declared that although a lot of techniques such as intermittent chemical cleaning, process optimisation and so on, had improved the membrane efficiency, but these techniques decreased net productivity and also generated secondary pollution. The authors also stated that the membrane fouling caused by organic matter would be converted into bio-fouling caused by extracellular enzyme produced by microorganisms and bacteria with time. This situation limits the usage of membrane system in water treatment purpose.

2.2 Heterogeneous Photocatalysis

2.2.1 Description

Generally, photocatalysis with titanium dioxide (TiO_2) is also known as one of the green technologies for its non-toxic, clean and safe properties. The photocatalysis is a very useful technology as is it is not only useful for water treatment, but also applicable in air purification, building exteriors, and other environmental cleanup. Heterogeneous photocatalytic oxidation process is an innovative water treatment technology because it is able to remove a wide range of pollutants and organic contaminants. In photocatalysis, pollutant is broken down into harmless substances such as carbon dioxide and water by the hydroxyl radicals generated from TiO_2 in the presence of UV light (Shon et al., 2008). Figure 2.3 shows the process of photocatalysis by degrading pollutants into harmless species.

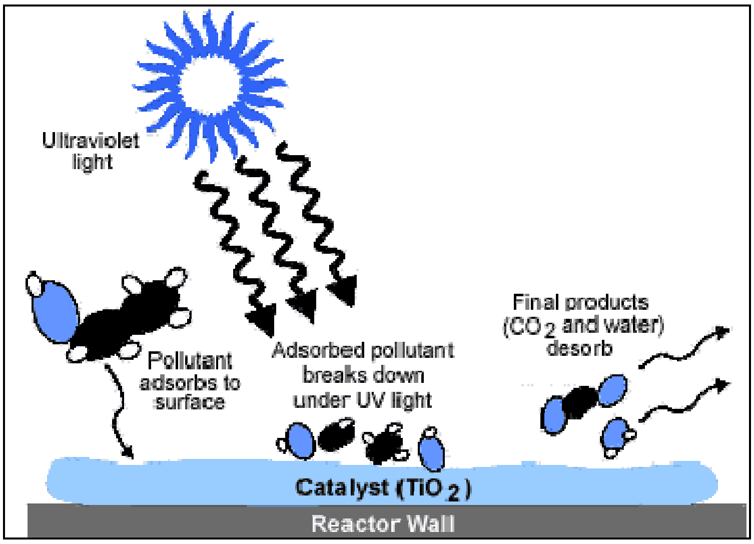


Figure 2.3: Process of Photocatalysis

2.2.2 Photocatalyst

Semiconductors are usually selected as photocatalysts as they have a narrow gap between the valence and conduction bands. For photocatalysis process, the semiconductors need to absorb energy equal to or more than its energy gap in order to activate the active site of the catalyst. Among the possible semiconductors, TiO_2 is usually chosen as the photocatalyst because of its low energy gap, which is 3.2 eV (Photocatalysis, 2005). Besides, TiO_2, especially the commercialized product named Degusa P25, is most extensively used because it has strong redox ability, chemical stability, non-environmental impact and low cost (Bhatkhande et al., 2001). The molecular structure of TiO_2 is shown in Figure 2.4.

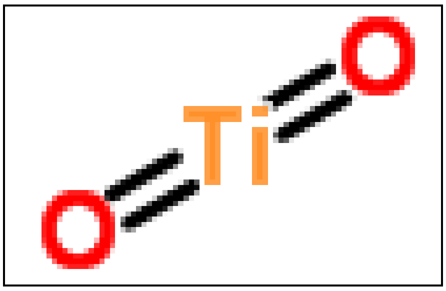


Figure 2.4: Molecular Structure of TiO_2

2.2.3 Light Source

According to Vigneswaran et al. (2009), the degradation of organic compounds by photocatalytic oxidation with UV light was much faster than that of the catalyst adsorption without UV light. Thus, UV light is important in order to achieve high efficiency of pollutant removal in photocatalysis. Figure 2.5 shows different types of UV light tubes mostly used in photocatalysis.

Anatase TiO_2, which is the most photoactive phase of TiO_2, only absorbs UV light with wavelength shorter than 380 nm. The most common used UV light source is the phosphor-coated black lamp (Type – c in Figure 2.5) due to its relative cheap price. Besides, the phosphor-coated black lamp also emits light in the UV-A bands (λ_{max} = 355 nm) which fulfils the requirement of anatase TiO_2 (Fujishima and Zhang, 2005). The black light tube is normally a fluorescent lamp with a different phosphor-coating which absorbs harmful short-wave UV-B and UV-C lights emitted from energised mercury atoms. Thus, it will only emit the long-wave UV-A light and some blue and violet lights. However, this light source produces relatively low power of UV, and thus it is not suitable for small-scale photocatalytic plant (Fujishima and Zhang, 2005).

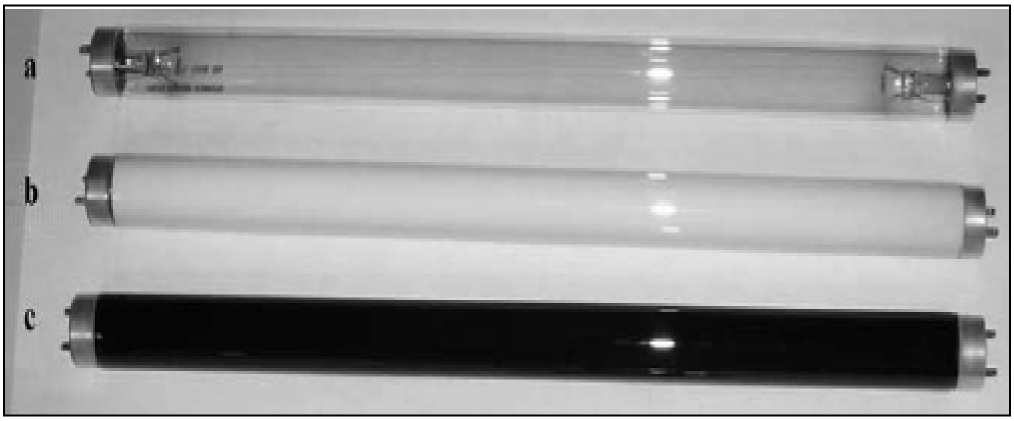


Figure 2.5: Different Types of UV Lamps. (a) Germicidal mercury lamp; (b) Phosphor-coated white lamp; (c) Phosphor-coated black lamp

2.2.4 Energy

As mentioned in Section 2.3.2, photocatalysis process will only occur when energy is sufficient to activate the active side of TiO_2. The heterogeneous photocatalytic process is initiated when a photon with energy equal to or greater than the energy gap band (E_{bg}) of the photocatalyst reaches the photocatalyst surface, resulting in molecular excitation. E_{bg} is defined as the difference between the filled valence band and the empty conduction band of the photocatalyst, in the order of a few electron volts (De Lasa et al., 2004). This molecular excitation generates mobile electrons in the higher energy conduction band and positive holes in the lower energy valence band of the catalyst, as shown in Equation 2.1.

$$\text{photocatalyst} \xrightarrow{E_{ph} \geq E_{bg}} e^- + h^+ \tag{2.1}$$

The movement of electrons forms negatively charged electron (e^-) or positively charged hole (H^+) pairs, as well as the formation of super oxides anions and hydrogen peroxide from oxygen. The hole can oxidize donor molecules (Photocatalysis, 2005). The reaction is illustrated in Figure 2.6.

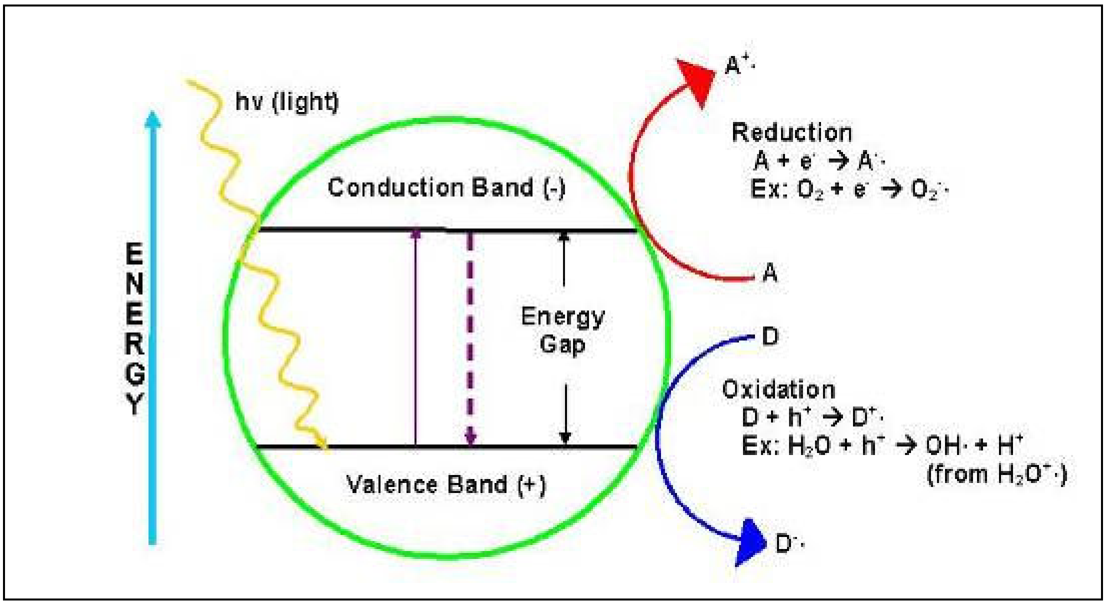


Figure 2.6: Reaction within a Catalyst under Light Source (Photocatalysis, 2005)

2.2.5 Chemical Reaction

According to De Lasa et al. (2004), the heterogeneous photocatalytic reaction involved a few mechanistic steps:

1. TiO_2 generates an electron and an electron-hole in the presence of UV light (Equation 2.1).

2. The electron transfers from the adsorbed surface (RX_{ad}), adsorbed water or the OH_{ad} ion, to the electron hole.

3. Water dissociates into ions and this produces high concentrations of OH^-. At the same time, oxygen molecule which acts as an acceptor species (O_2^-) reacts with H^+ and $HO_2^\bullet$ forms hydrogen peroxide (H_2O_2). Photoconversion of hydrogen peroxide gives more $OH^\bullet$ free radical groups.

4. The $OH^{\bullet}$ radicals oxidize organic adsorbed pollutants (RX_{ad}) onto the surface of the TiO_2 particles. The $OH^{\bullet}$ radicals is very reactive and attack the pollutant molecule (as shown in Equation 2.2) to degrade it into mineral acids including carbon dioxide and water.

$$OH^{\bullet}_{ad} + RX_{ad} \rightarrow \text{Mineral acid} \qquad (2.2)$$

2.2.6 Advantages

Photocatalysis provides many advantages over the world, especially in the aspect of environment. Due to its high efficiency and the generation of harmless by-products, it is one of the green technologies that catch the attention of scientists in the world. It is widely used because of its self-cleaning, air cleaning, water purification, antitumor activity, self-sterilizing properties (Kaneko and Okura, 2002). These properties can be applied to protect the wall of buildings, concrete, cement, tiles and also sterile equipments in hospital's operating rooms. Besides, photocatalysis can eliminate polluted compounds in air such as nitrogen oxides (NO_X) (Figure 2.7), cigarette smoke, as well as volatile compounds arising from various construction materials (Anpo, 2000). Other applications of photocatalysis are illustrated in Figure 2.8.

Figure 2.7: View of soundproof highway walls coated with TiO_2 photocatalysts for the elimination of NO_X

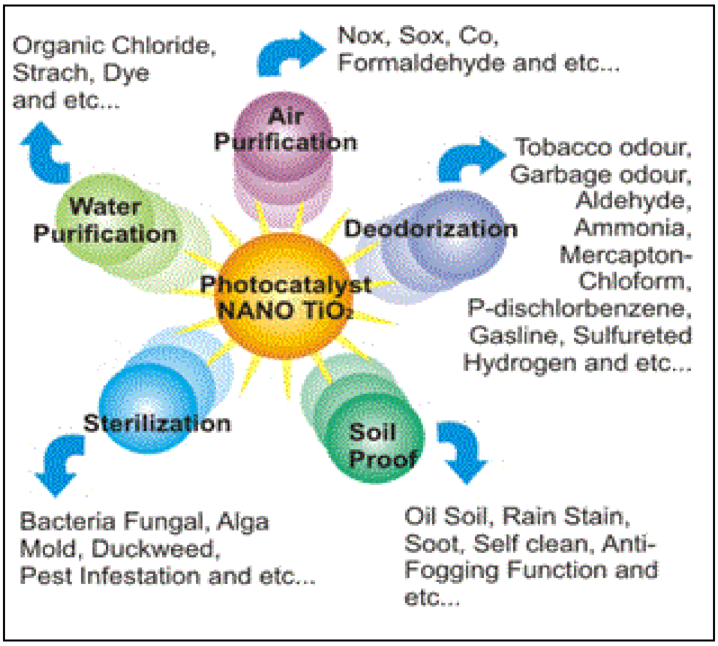


Figure 2.8: Applications of Photocatalysis Technology

2.2.7 Disadvantages

In photocatalytic processes, the catalyst is usually applied in the form of powder suspended in slurry. However, there is a problem with this kind of approach in large scale, which has difficulty in recovery of catalyst from the solution at the end of operation. It is important to separate the catalyst particles with treated water before the exit of treatment plant. Although this problem can be solved by immobilizing the catalyst on an inert surface, such as glass, quartz, concrete or ceramics, but this will limit the activity of the catalyst and decrease efficiency of the operation (Augugliaro et al., 2006). Thus, solid-liquid separation knowledge is needed to be applied in the system in order to prevent a decrease of the catalyst concentration in it and avoid wash out of the catalyst which may cause water pollution.

2.3 Integrated Photocatalysis-Membrane Hybrid System

2.3.1 Advantages

An integrated photocatalysis-membrane hybrid system is the combination of photocatalysis reactor and a membrane to filter treated water with catalysts. This hybrid system provides a near-zero fouling system. According to Shon et al. (2008), if UV was irradiated directly on the membrane surface, the photocatalytic reaction could convert the foulant on membrane surface, which consists of aromatic organics to aliphatic compounds, resulting in the reduction of the membrane fouling. In addition, photocatalysis is the pre-treatment to remove organic foulants before the water deliver to the membrane for final treatment. This step not only reduces membrane fouling, but also increases the permeate flux of the membrane. The membrane separates treated water with catalyst and acts as a barrier in recovering the catalyst. Thus, this hybrid system can overcome problems encountered in the membrane filtration and heterogeneous photocatalysis technology.

2.3.2 Researches on Photocatalysis-Membrane Hybrid System

Xi and Geissen (2001) investigated the possibility of catalyst separation from water by cross-flow microfiltration by using suspensions of TiO_2 and thermoplastic membranes. The study shows that the separation performance TiO_2 of particles is strongly affected by feed concentrations, pH and ionic strength, cross-flow velocity and transmembrane pressure. An extreme sensitivity to pH and electrolyte concentration shows the importance of interfacial effects on solid-liquid separation of TiO_2 particles. The results obtained in the study made TiO_2 separation by cross-flow microfiltration attractive in solar-catalytic detoxification.

In addition, a study of determine the possibility of coupling photocatalysis and membranes for degradation of fulvic acid is investigated by Fu et al. (2006). They used a submerge membrane photocatalytic reactor with a nano-structured TiO_2 silica gel photocatalyst. The material set up of this photoreactor is shown in Figure

2.9. The catalyst particles were small enough to form a homogeneous suspension and big enough to easily settle. Then, the microfiltration separation process allowed the TiO_2 to be easily separated, recovered and reused. Thus, this maintains the high flux of the membranes. The authors declared that according to their experiments, the permeate flux rate of the membrane is improved and thus the membrane fouling phenomenon is reduced with the addition of nano-structured TiO_2 catalyst. Therefore, the submerged membrane photocatalysis reactor can be potentially applied in photocatalytic oxidation process during drinking water treatment.

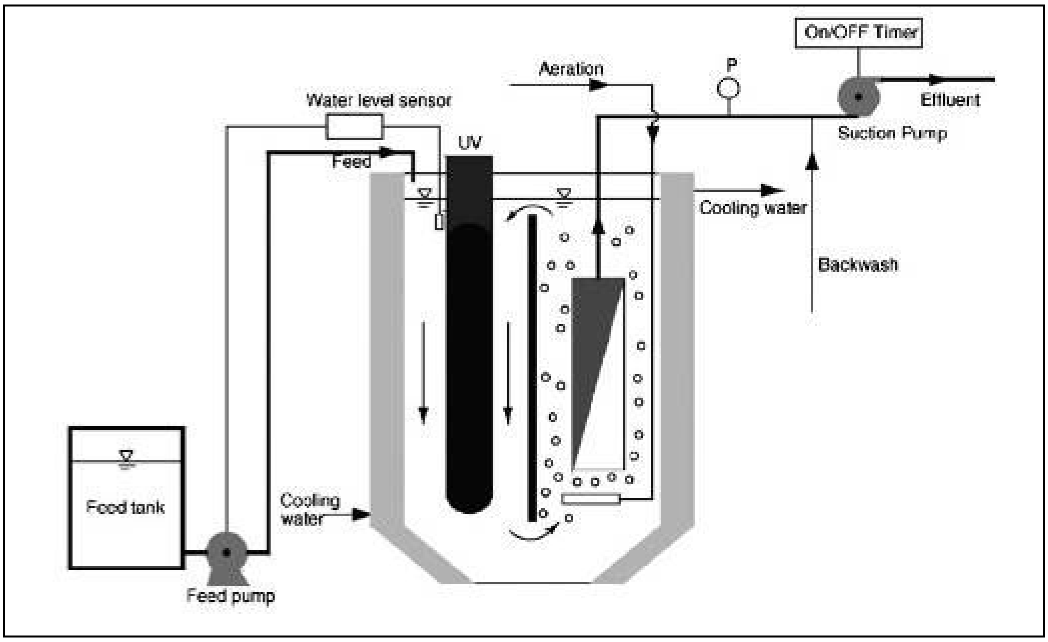


Figure 2.9: Diagram of Submerged Membrane Photocatalytic Reactor for Fulvic Acid Removal (Fu et al., 2006).

2.4 Cyanide

2.4.1 Description

Cyanide is highly toxic with its cyano group (C≡N). Various forms of cyanide ions (CN^-) exist, including gaseous hydrogen cyanide (HCN), water-soluble potassium cyanide (KCN) and sodium cyanide (NaCN) salts, and other poorly water-soluble mercury, copper, gold, and silver cyanide salts. HCN is a pale blue or colourless liquid at room temperature and colourless gas at higher temperature condition. It has a bitter almond odour. KCN and NaCN are white powder with a bitter almond-like odour (The Facts about Cyanides, 2004). Although cyanide compounds have a characteristic odour, odour is not a good way to tell if cyanide is present.

After the exposure of cyanide, it quickly enters the bloodstream. If there is small amount of cyanide in the bloodstream, body can change cyanide into thiocyanate, which is less harmful and excreted in urine. On the other hand, if large amount of cyanide enters the body, the body will be unable to change all of the cyanide into thiocyanate. Large doses of cyanide in bloodstream prevent cells from using oxygen and causing body cells to die (The Facts about Cyanides, 2004).

2.4.2 Regulatory Limits

In order to protect the environment and water bodies, effluents containing cyanide from various industries must be treated before releasing into the environment. Hence, many countries and environmental protection agencies impose their own limiting standards for discharge of cyanide. The United States Environmental Protection Agency (US EPA) had proposed a limit for drinking waters regarding total cyanide are 200 ppb, where total cyanide refers to free and metal-complex cyanides (US EPA., 1985). The German and Swiss regulations had set limit of 0.5 mg/L for cyanide in sewers and 0.01 mg/L for cyanide in drinking water (Parga et al., 2003). The Central Pollution Control Board (CPCB) in India had also set a minimal national standard (MINAS) limit for cyanide in effluent as 0.2 mg/L (Dash et al., 2008). Lastly, the Ministry of Health Malaysia had set limit of 0.2 mg/L for sewers and 0.07 mg/L in drinking water (Ministry of Health Malaysia, 1983). Nevertheless, these regulatory limits had become one of the considerations on water treatment for the removal of cyanide. It is necessary to lower the concentrations of cyanides to below the regulatory limits.

2.5 Cyanide Removal Methods

2.5.1 Chlorination Process

The most common method to remove cyanide is the chemical oxidation techniques such as the chlorination process. Wastewater which contains cyanide is initially treated with chlorine or hypochlorite to produce cyanogen chloride, which then reacts to form the less toxic sodium cyanate. Further chlorination process will oxidise the cyanate to carbon dioxide and nitrogen.

Although this method of treatment is easy and efficient in detoxifying free cyanide in wastewater, it has several disadvantages. Chlorination is not useful when the cyanide in wastewater bonded with metals such as nickel, silver, and so on (Patil et al., 2000). The process is also relatively expensive due to the quantity of chlorine required. In addition, the excess chlorine increases the total solids content of water, causing the water unable to use for recycling and reuses purposes. Besides it leaves a residue with high chlorine content which is toxic to aquatic life (Kao et al., 2003) and other living organisms as it can destroys living tissue (Gáspár, 2000).

2.5.2 Bio-treatment Process

Cyanide in water can be treated using bio-treatment process. The biodegradation of cyanide under anaerobic conditions has also recently demonstrated the feasibility to biogas generation, a possibly economic benefit of the process (Ebbs, 2004). Significant advances have been reported in the use of plants to remove contaminants in water, including cyanide compounds (Aksu et al., 1999). Degradation of cyanide by microorganisms from wastewater process is a proven as an alternative to traditional chemical and physical processes (Kao, 2006).

The microorganisms for cyanide degradation process can be generally divided into two classes, which are bacteria and fungus. Type of microorganisms used in the process depends on the type of reactor, type of cyanide species inside the water and other water conditions (Dash et al., 2008). There are a several suspended growth processes available for the aerobically biological treatment of cyanide and thiocyanate. The microorganisms involved in the biological treatment of cyanide and thiocyanate are usually taken from a heterogeneous mixture, such as indigenous soil bacteria because the microorganisms is adapted to the environment and able to function well during treatment of these compounds (Mudder et al., 2001). However, the thiocyanate in water is not easy to remove through anaerobic biological treatment. In addition, this process requires higher capital cost. The oxidation processes for cyanide degradation in settling ponds are expensive and also cause environmental problems (Desai and Ramakrishna, 1998).

2.5.3 Activated Carbon Technology

Granular or powered carbon is the most widely used adsorbent, as it has a good adsorption on organic and inorganic molecules. An activated carbon particle, whether in a powdered or granular form, has a porous structure which consists of inter-connected macropores and mesopores networks that provide a good capacity for the adsorption due to its high surface area (Hanaki et al., 1997). Activated carbon is effective for the oxidation of cyanide. It acts as an adsorbent and also as a catalyst for the oxidation process of cyanide. Adsorption technique is a widely used technology for the removal and recovery of cyanide (McKay and Bino, 1987).

In the process, the activated carbon is added into the wastewater and air sparger is required in order to introduce air molecules into the solution. Then, cyanide will oxidise to cyanate in the presence of activated carbon (Adams, 2003). Although activated carbons have greater quality of removal of cyanides from industrial wastewaters, it has never been used as a main treatment method. This is because it is costly, require good treated activated carbon, used only for low concentrations of cyanide, pretreatment required, and also requires further treatment (Dash et al., 2008).

2.5.4 Gas-Filled Membrane Technology

Another method to remove cyanide is using gas-filled membrane. Han et al. (2005) performed a research on using hollow fiber gas membranes in a pilot plant to remove cyanide from four industrial wastewaters. Figure 2.10 is the pilot plant diagram used in their research. A hydrophobic microporous membrane, such as polypropylene (PP) or polytetrafluoroethylene (PTFE), was used to separate the two aqueous streams. Hydrogen cyanide (HCN) transferred through the gas-filled hydrophobic microporous membranes to striping solution containing sodium hydroxide (NaOH).

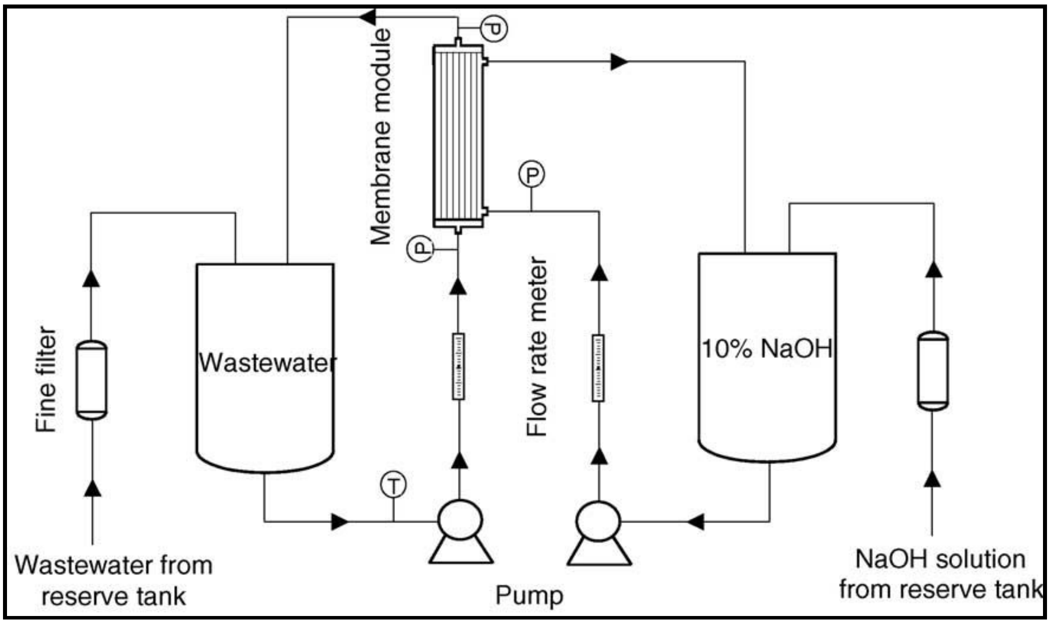


Figure 2.10: Pilot Plant Diagram of Cyanide Removal Using Gas Membrane (Han et al., 2005).

The schematic diagram of a gas-filled microporous membrane for cyanide removal involved inside the process is illustrated in Figure 2.11. According to Han et al. (2005), the membrane pores remain gas-filled with condition where the pressure difference between the two aqueous phases is less than the breakthrough pressure. The wastewater containing cyanide flows on one side of the membrane, while the reactive strip solution containing sodium hydroxide (NaOH) flows on the other side with opposite direction. Hydrogen cyanide (HCN) from water has properties such as weak acid and high volatile. It will vaporise and diffuse across the gas-filled pores and then into the strip solution. In the reactive strip solution, the reaction of forming sodium cyanide (NaCN) is shown in Equation 2.3.

$$HCN + NaOH \leftrightarrow NaCN + H_2O \qquad (2.3)$$

The reaction between HCN and NaOH is very rapid, thus the HCN concentration in the strip solution is approximately equal to zero. Consequently, HCN will continue to transfer from the feed to the strip solution providing as long as excess base is present in the strip solution. According to Han et al. (2005), the use of gas-filled membrane pores provides some advantages, such as cyanide can be recovered and reused, no secondary pollutants are produced, the energy and chemical requirements are low and the equipment is simple to operate.

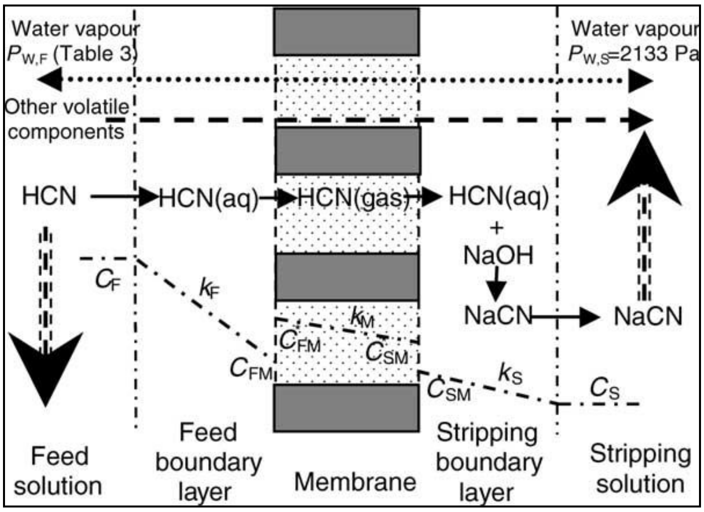


Figure 2.11: Illustration of HCN removal by gas membrane absorption (Han et al., 2005).

2.5.5 Photocatalysis Oxidation

The kinetics and mechanism of the photocatalytic oxidation of cyanide by using TiO_2 were investigated by Chiang et al. (2003). By displacing the surface hydroxyl groups on the surface of TiO_2 with fluoride ions, cyanide is oxidized by the holes trapped at the surface hydroxyl groups. The authors declared that the quantum efficiency of the photocatalytic oxidation was found to be low due to several reasons, such as low adsorption of cyanide ions (CN^-) onto the TiO_2 surface, the absence of homogenous reaction between CN^- and diffused hydroxyl radicals, and the high electron hole recombination rate within the TiO_2 photocatalyst. In addition, a kinetic model was developed in their study to describe the mechanism involved in the photocatalytic oxidation of cyanide.

According to the authors, cyanate (OCN^-) is found to be the oxidation product which can be further oxidised on the surface of TiO_2 to produce nitrate and carbonate. Other than oxidation, the hydrolysis of cyanate is also possible in this study. By using hydrolysis, the process can produce ammonium and carbonate as shown in Equation 2.3 and 2.4.

$$CN^- + 2OH^- + 2h^+_{vb} \xrightarrow{TiO_2+UV} OCN^- + H_2O \tag{2.4}$$

$$OCN^- + 2H_2O \rightarrow +NH_4^+ + CO_3^{2-} \tag{2.5}$$

Furthermore, the effect of organics on cyanide oxidation is studied by Osathaphan et al. (2008). The organic ligand used in the study is ethylenediaminetetraacetate (EDTA), which can forms strong complex with heavy metal ions. According to the study, the presence of EDTA in the reaction mixture reduces the photocatalytic oxidation rate of cyanide. The authors further explain the photocatalytic oxidation of cyanide in absence and presence of EDTA by proposed a mechanism.

2.5.6 Other Methods

There are many other methods used to remove cyanide from water. Table 2.1 shows the summary of some of the methods and their problems.

Table 2.1: Other Cyanide Removal Methods

Method	Description	Problem
Sulfur oxidation **(Han et al., 2005)**	Cyanide is oxidized to cyanate using sulphur dioxide or ferrous sulphate and air in the presence of copper ions. Sulphuric acid formed is neutralized with lime.	Reaction is slow at low temperature, unable to remove thiocyanate, cyanate, or ammonia, cyanate can transformed into toxic ammonia by microbial action and reaction requires high energy.
Hydrogen peroxide **(Lee et al., 2004)**	Cyanide oxidised to cyanate using hydrogen peroxide in the presence of copper ion.	Hydrogen peroxide is hazardous and expensive, requires specialized equipment to increase the total capital cost of the plant, the process generates ammonia which is toxic to aquatic and requires accurate measurement of chemical dose.
Ozonation **(Carrillo-Pedroza et al., 2000)**	Cyanide is oxidised using ozone.	Produce ammonia, reagent and equipment of the process are costly, unable to in the removing of iron-cyanide complex, need extra treatment process.
Acidification-volatilisation-recovery (AVR) **(Mosher and Figueroa, 1996)**	The pH of a cyanide solution is lowered by adding sulphuric acid to form HCN gas. Then the gas will absorb into NaOH solution.	More complex and hazardous than other process, sealed mixing vessel and packed columns are require, and economy value depending on the total recovery of cyanide from the process.
Ion exchange **(Fernando et al., 2002)**	Cyanide is removed by anion exchange resin.	Regeneration of resin is difficult since there are cyanide complexes besides free cyanides.

CHAPTER 3

METHODOLOGY

3.1 Project Planning

The project's schedule was planned at the beginning of the project. This is an important procedure in every single project to identify necessary adjustment in the remaining plans to meet the due date. The important tasks of the project were listed down and planned by using Gantt chart in Microsoft Office Project. Gantt chart is a useful planning tool as it is easily understood and provides a picture of the current state of the project. The Gantt chart of this project is shown in Appendix A. The progress of the project was updated each week in order to ensure the actual progress was following the track of planned progress.

3.2 Materials

The photocatalyst used in this study was P25 Degussa TiO_2 powder. Its properties are shown in Table 3.1 adapted from Shon et al. (2008).

Table 3.1: Properties of TiO_2 Photocatalyst

Properties	Specification
Structure	Non-porous
Components	80% anatase, 20% rutile
Primary Crystal Size	3 µm
Apparent Density	130 kg/m^3
Surface Area	42.32 ± 0.18 m^2/g
Type	Powdered
Product Code	Degussa P25

Synthetic wastewater with an appropriate amount of cyanide was used in this experiment. The pH of the wastewater was adjusted to pH 12.0 by adding 1 M of sodium hydroxide (NaOH) into 4 L of Milli-Q water, followed by adding the desired volume of potassium cyanide (KCN). The pH adjustment is important because volatility losses of free cyanide, as HCN, can occur in samples with pH values less than 11 (Method 9016, 2010).

Moreover, an ultrafiltration (UF) hollow fiber membrane was used in this study. The main function of the membrane is to perform the solid-liquid separation of treated water and photocatalyst. The membrane was washed using mild acid, then soaked in mild alkaline and lastly rinse with Milli-Q water.

Four phosphor-coated UV lamps (from Sankyo Denki Co., Ltd., Japan, type F8T5BLB) were used to activate the photocatalyst in the experiment. The UV lamps were 4 Watt (W) each and could emit 352 nm of radiation, encased within transparent glass tube. These UV lamps were placed inside the feed tank together with TiO_2 photocatalyst.

3.3 Photocatalysis Membrane-Hybrid System Set Up

The hybrid system set up is shown in Figure 3.1. This system consisted of a reactor tank (around 6 litre), reverse osmosis booster pump, an ultrafiltration (UF) type of hollow fiber membrane unit and a product tank (around 10 litre). Synthetic wastewater which contained cyanide was transferred into the reactor tank, followed by adding an amount of TiO_2. A mechanical stirrer and UV lamps were turned on to start up the reactions. After a certain period, the treated water were pumped to the membrane unit and then to the product tank.

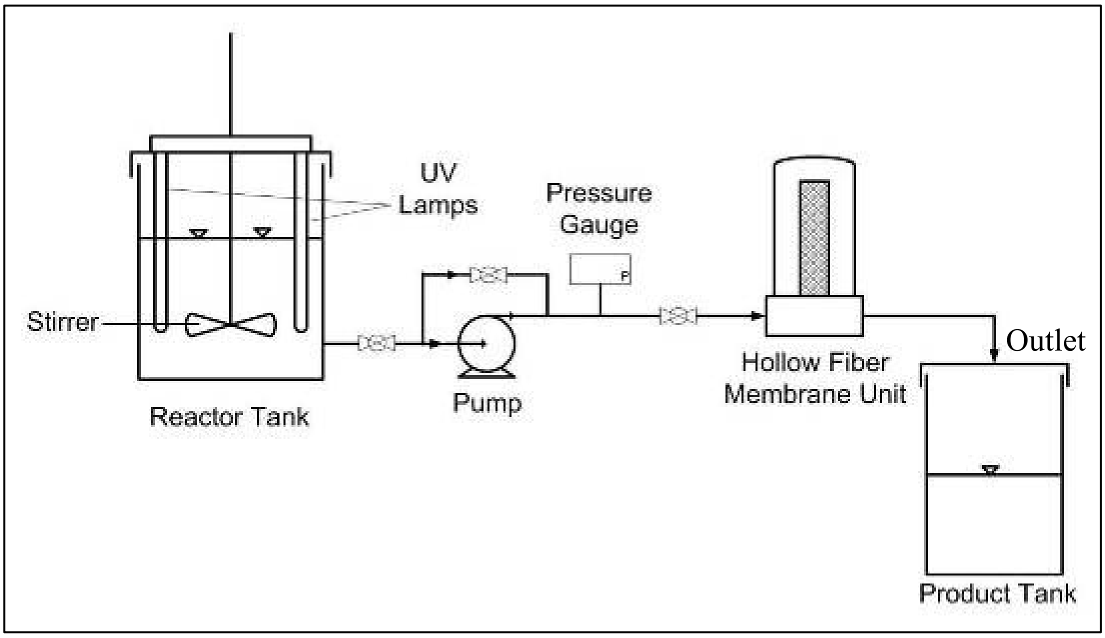


Figure 3.1: Schematic Drawing of Photocatalysis-Membrane Hybrid Unit

3.4 Chemical Hazards, Safety and Precautions

3.4.1 Chemical Hazards

Chemicals used in this experiment can be hazard if mishandled. Chemicals safety data is referred to the Material Safety Data Sheet (MSDS) in Appendix B, obtained from Chemical Safety Data: Potassium Cyanide (2005) and Sodium Hydroxide (2006). Below are the information obtained:

1. (1) Potassium cyanide (KCN) is highly toxic and can cause fatal if swallowed. Large amount of KCN that is absorbed through the skin can also cause serious harm and fatal. Skin contact with KCN may lead to burns. Moreover, KCN reacting with acid generates hydrogen cyanide (HCN) gas which is extremely toxic.
2. (2) Sodium hydroxide (NaOH) solid and solution are corrosive. Spill of NaOH can cause apparatus damage. Besides, contact of NaOH with skin can cause burn and contact with eyes can cause serious long-term damages.

3.4.2 Safety Precautions

Some safety precautions steps must be followed during performing the experiments. The steps are as follows:

- Always wear safety glasses, mask and gloves when handling chemicals. Avoid the solution to come into contact with skin or eyes.
- If any chemicals come into contact with the skin, rinse off with plenty of water immediately.
- Ensure proper ventilation in laboratory during working on the experiments.
- Wash away any spill of chemicals immediately.
- Dispose all unused chemicals in an appropriate manner after the experiments. Do not allow the chemicals flow into sinks or drains.
- Avoid eyes on UV lamp for long period as it can cause blind.
- Wash hands with soap after the experiments.

3.5 Preparation

Basic information of the module, such as reactor height, reactor volume, working volume, light intensity, water temperature and water pH were recorded before starting the experiments. The pH and temperature of the water in this study were measured using pH meter (Eutech Instruments Cyberscan Model pH 300).

Preliminary test was performed at the beginning of the experiments in order to ensure that the pure Milli-Q water was free of any oxidising agents that could decompose cyanide and affect the results. Moreover, the initial concentration of cyanide in the pure Milli-Q water was also determined.

Before any experiment started, a standard solution was prepared. First of all, water with pH 12.0 was prepared by adding 1 M of NaOH into 6 L of Milli-Q water. Next, 1 L of the water was used to prepare cyanide stock solution with an approximate of 50 mg/L CN^- by adding 0.125 g of KCN. After the stock solution was ready, the other 5 L of water was used to prepare five different CN^- concentrations by adding different volumes of stock solution into 1 L water each. Based on the results obtained, a standard calibration curve of cyanide volume was produced. Hence, the desired concentration of cyanide for the following experiments could be easily determined.

3.6 Experiments

3.6.1 Experiment A: Investigate the Effect of Different TiO_2 Concentrations

This experiment was to investigate the effect of TiO_2 concentration on the cyanide removal efficiency and the first-order rate constant using the photocatalysis-membrane hybrid system. Synthetic wastewater with the concentration of 0.2 mg/L CN^- and four UV lamps were used in this experiment as the control variables. The synthetic wastewater was kept in the reactor and then added an amount of TiO_2. Below are the procedures involved:

1. Four litres of synthetic wastewater with 0.2 mg/L CN^- was transferred into the reactor tank.
2. The solution was added with 8.0 g of TiO_2 (2.0 g/L TiO_2), then pump was turned on for 15 seconds to allow the solution to pass through the membrane. A 40 mL of water sample was collected from outlet as initial sample.
3. UV lamps and stirrer in the reactor were switched on to start the reactions.
4. Forty millilitres of water samples were collected at the reaction time of 30 minutes, 1 hour, 2 hours, 3 hours, 4 hours, 5 hours and 6 hours. Before the samples were collected, pump was turned on for 10 seconds to ensure that the solution from reactor completely passed through the membrane.
5. Water samples were collected for analytical tests and the removal efficiency and the rate constant were calculated.
6. Steps 1 to 5 were repeated using 1.5, 1.0, and 0.5 g/L of TiO_2.

3.6.2 Experiment B: Investigate the Effect of UV Light Power

This experiment was to investigate the effects of UV light power on the cyanide removal efficiency and the first-order rate constant using photocatalysis-membrane hybrid system. A 4 L of synthetic water with 0.2 mg/L CN^- was used as the feed of this experiment. The best performance of TiO_2 amount in Experiment A was chosen in this experiment. Both of the 0.2 mg/L CN^- synthetic water and chosen TiO_2 concentration were control variables in this experiment. Below are the procedures involved:

1. Four litres of synthetic wastewater with 0.2 mg/L CN^- was traanferred into the reactor tank.
2. The solution was added with chosen TiO2 amount (1.0 g/L TiO2, based on results in Experiment A). The pump was turned on for 15 seconds to allow the solution to pass through the membrane. Forty millilitres of water sample was collected from outlet as initial sample.

3. Four UV lamps and stirrer in the reactor were switched on to start the reactions.
4. Forty millilitres of water samples were collected at the reaction time of 30 minutes, 1 hour, 2 hours, 3 hours, 4 hours, 5 hours and 6 hours. Before the samples were collected, pump was turned on for 10 seconds to ensure that the solution from reactor completely passed through the membrane.
5. Water samples were collected for analytical test and the removal efficiency and the rate constant were calculated. Besides, the light intensity was also calculated.
6. Steps 1 to 5 were repeated using three lamps, two lamps, one lamp, and no lamp.

3.6.3 Experiment C: Investigate the Effect of Cyanide Concentration

This experiment was to investigate the effect of different initial cyanide concentrations on the removal efficiency and rate constant using the photocatalysis-membrane hybrid system. The best performance of TiO_2 concentration and UV intensity were chosen in Experiments A and B, respectively. Both of the TiO_2 concentration and UV light intensity were used as control variables in this experiment. Below are the procedures involved:

1. Four litres of synthetic wastewater with 0.3 mg/L CN^- was poured into the reactor tank.
2. The solution was added with chosen TiO_2 amount (1.0 g/L TiO2, based on results in Experiment A). The pump was turned on for 15 seconds to allow the solution to pass through the membrane. Forty millilitres of water sample was collected from outlet as initial sample.
3. Chosen amount of UV lamps (four UV lamps, based on results in Experiment B) and stirrer in the reactor were switched on to start the reactions.
4. Forty millilitres of water samples were collected at the reaction time of 30 minutes, 1 hour, 2 hours, 3 hours, 4 hours, 5 hours and 6 hours. Before the

samples were collected, pump was turned on for 10 seconds to ensure that the solution from reactor completely passed through the membrane.

5. Water samples were collected for analytical test and the removal efficiency and rate constant were calculated.
6. Steps 1 to 5 were repeated using 0.2, 0.15, and 0.1 mg/L CN^-.

3.7 Total Recovery of Photocatalyst

The concentration of TiO_2 remaining in the treated water was determined by collecting the total suspended solid (TSS) contained in each water samples. Average TSS from permeate of each experiment was calculated. With these data, the total recovery of photocatalyst in each experiment were determined.

3.8 Analytical Test

3.8.1 Method

The method used in this study to determine the amount of cyanide found in water is pyridine-pyrazalone method (Method 8027) using a VIS spectrophotometer (Hach Model DR2800). Besides, the TSS was lso tested using the same spectrophotometer. The spectrophotometer used in the study is shown in Figure 3.2.

Figure 3.2: Hach DR2800 Spectrophotometer

3.8.2 Apparatus and Materials

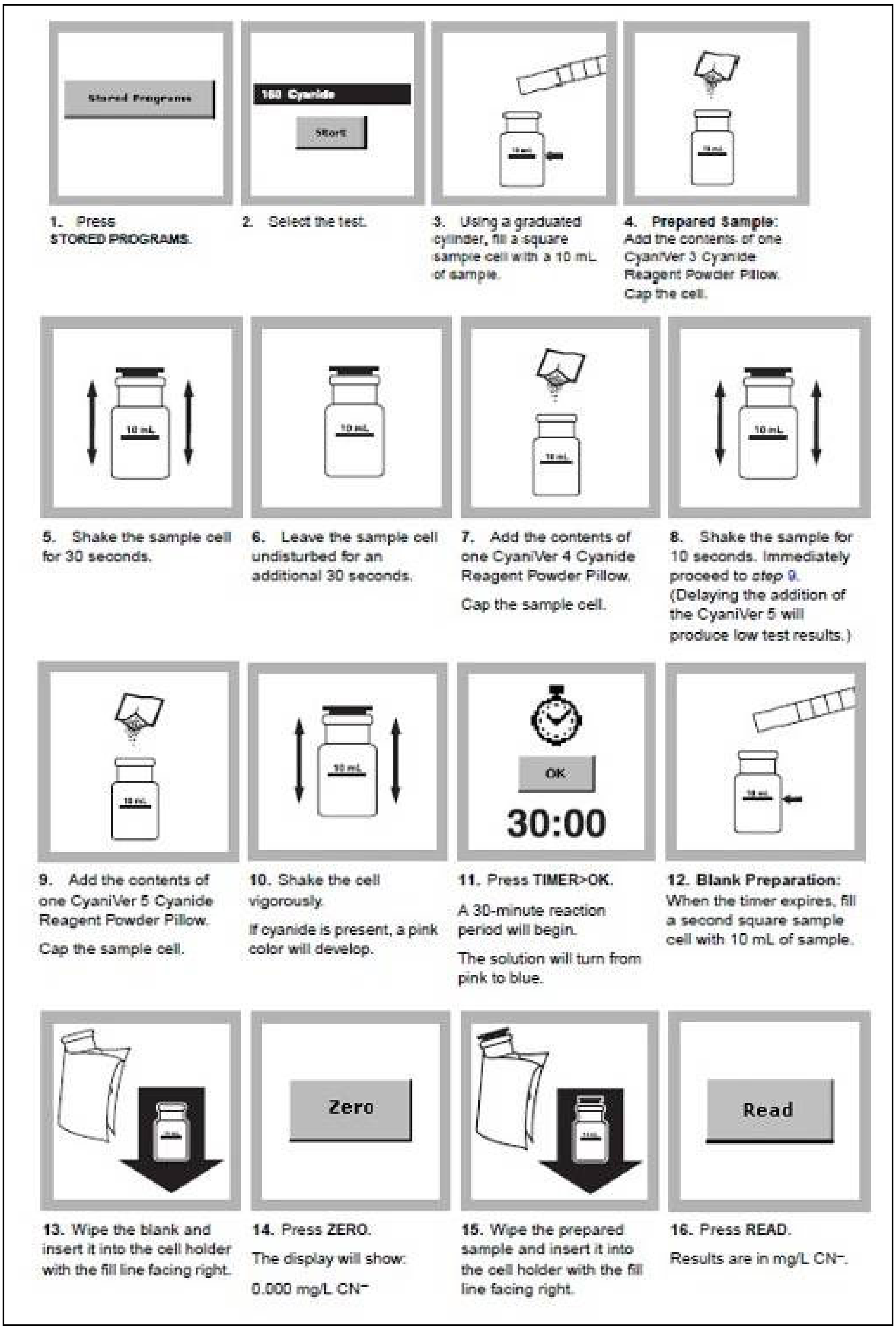
The following apparatus and materials were prepared for the analytical test of the samples obtained from experiments:

- 2 M of hydrochloric acid (HCl)
- CyaniVer® Cyanide 3 Reagent Powder Pillow
- CyaniVer® Cyanide 4 Reagent Powder Pillow
- CyaniVer® Cyanide 5 Reagent Powder Pillow
- Cylinder
- 1-inch square glass sample cells

3.8.3 Procedures

The procedures to determine total cyanide concentration in water sample followed the procedure manual of spectrophotometer. Water sample from the experiment was adjusted to pH 7.3 with 2 M of HCl under fume hood. This step is to release the CN^- which is attached to the alkali (NaOH) during the experiment. After the pH adjustment, water sample was filled into the sample cells to the level of 10 mL. Then, CyaniVer 3 Cyanide Reagent Powder Pillow was added into the sample cell and shook for 30 seconds, followed by waiting for an additional of 30 seconds. Next, CyaniVer 4 Cyanide Reagent Powder Pillow was added into the cell and shook for 10 seconds. CyaniVer 5 Cyanide Reagent Powder Pillow was added immediately, shook and left aside for 30 minutes for reactions. At the beginning of the reaction, a pink colour solution was developed, indicating the presence of cyanide. After the reaction, the solution turned from pink to blue colour as shown in Figure 3.4. Another sample cell was used to prepare a blank sample using Milli-Q water. Blank sample was used to set zero value, whereas the water sample after reaction was collected to read using spectrophotometer. The complete procedure of this analytical test is shown in Figure 3.3, obtained from DR2800 Spectrophotometer Procedures Manual (Edition 1).

For total suspended solid (TSS) analytical test, Milli-Q water and the water sample from experiment were transferred into the 10 mL sample cell. The Milli-Q water was used to set zero value in the spectrophotometer before water sample was tested for the TSS.



1. Press **STORED PROGRAMS**.

2. Select the test.

3. Using a graduated cylinder, fill a square sample cell with a 10 mL of sample.

4. **Prepared Sample:** Add the contents of one CyaniVer 3 Cyanide Reagent Powder Pillow. Cap the cell.

5. Shake the sample cell for 30 seconds.

6. Leave the sample cell undisturbed for an additional 30 seconds.

7. Add the contents of one CyaniVer 4 Cyanide Reagent Powder Pillow.

Cap the sample cell.

8. Shake the sample for 10 seconds. Immediately proceed to *step 9*. (Delaying the addition of the CyaniVer 5 will produce low test results.)

9. Add the contents of one CyaniVer 5 Cyanide Reagent Powder Pillow.

Cap the sample cell.

10. Shake the cell vigorously.

If cyanide is present, a pink color will develop.

11. Press **TIMER>OK**.

A 30-minute reaction period will begin.

The solution will turn from pink to blue.

12. **Blank Preparation:** When the timer expires, fill a second square sample cell with 10 mL of sample.

13. Wipe the blank and insert it into the cell holder with the fill line facing right.

14. Press **ZERO**.

The display will show:

0.000 mg/L CN⁻

15. Wipe the prepared sample and insert it into the cell holder with the fill line facing right.

16. Press **READ**.

Results are in mg/L CN⁻.

Figure 3.3: Procedures Manual Cyanide Test Using Spectrophotometer

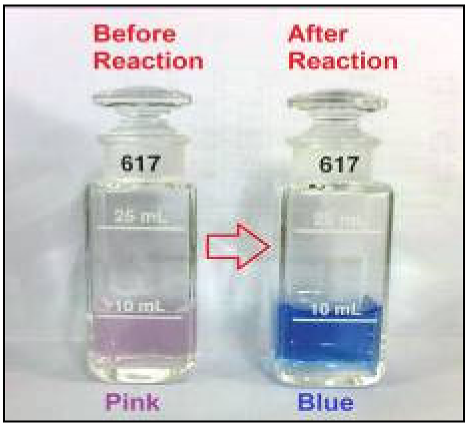


Figure 3.4: Before and After Reaction of Water Sample

3.9 Data Analysis

Removal efficiency of cyanide was calculated by Equation 3.1. Final concentration of sample was obtained by calculating the mean value from the readings recorded from experiment.

$$\eta = \left(1 - \frac{C}{C_o}\right) \times 100\% \tag{3.1}$$

where

η = removal efficiency (%)

C_o = initial concentration (mg/L) of cyanide in sample ($t = 0$)

C = concentration (mg/L) of cyanide in sample at time, t

The experimental data were examined to see if they fitted the First-order Kinetics Model (Equation 3.2). The equation was further derived (Equations 3.3, 3.4 and 3.5) in order to obtain the rate constant, k from the graph of $\ln(C/C_o)$ versus time.

$$r = \frac{\mathrm{d}C}{\mathrm{dt}} = -kC \tag{3.2}$$

$$\int_{C_o}^{C} \frac{1}{C} dC = -k \int_{0}^{t} dt \tag{3.3}$$

$$\ln\left(\frac{C}{C_o}\right) = -kt \tag{3.4}$$

$$\ln\left(\frac{C_o}{C}\right) = kt \tag{3.5}$$

where

r = rate of photo-degradation of cyanide (mg/L-h)

k = first-order rate constant (h^{-1})

t = time (h)

The linear regression performed using Data Analysis function in Microsoft Excel was statistically significant when the p-value was below 0.05.

Total recovery of photocatalyst was calculated using Equation 3.6.

$$R = \left(1 - \frac{C_P}{C_F}\right) \times 100\% \tag{3.6}$$

where

R = Total recovery of TiO_2

C_F = Concentration of TiO_2 in feed

C_P = Average concentration of TiO_2 in permeate

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Experimental Preparation

4.1.1 Tank Modification

Four holes were made on the top cover of the original reaction tank's (reactor) to place the UV lamps. Besides, a non-transparent material was used to cover the body of the reactor to prevent the emission of UV list to the surrounding environment. A lamp supporting tool was designed and installed on the top of the reactor to hold the lamps tightly inside the reactor, so that the lamps did not contact with the interior of the reactor, and the stirrer. The modified top cover and lamp supporting tool are shown in Figure 4.1.

Figure 4.1: Modified Reactor

4.1.2 Basic Information

The basic information of reactor and operating conditions of this study are summarized in Table 4.1. These figures were established based on the data and calculations from Appendix C. In addition, the power of UV lights used was shown in Table 4.2.

Table 4.1: Basic Information of Reactor and Operating Condition

Features	Specifications	Values
Reactor	Height, H_T	26.1 cm
	Diameter, D_T	17.1 cm
	Volume, V_T	6.01 L
Operating/ working	Water level, H_W	20.0 cm
	Diameter, D_W	16.2 cm
	Volume, V_W	4.00 L
	Temperature	23°C
	pH	12.0
	Flow rate (First 15 s)	0.584 LPM
	Flow rate (Following time)	0.900 LPM

Table 4.2: Power of UV Light

Number of Lamp	Power (W)
0	0
1	4
2	8
3	12
4	16

4.1.3 Prelimary Test

At the early stage of this study, the Milli-Q water was tested to ensure that there were no oxidising agents and cyanide in the water. Table 4.3 shows that some compounds tested in the test. The result showed that the water did not contain any chloride, sulphate, cyanide and total suspended solids. According to the Millipore Company (2010), Milli-Q water is ultrapure water in which most of the contaminants are removed by the combination of different water purification techniques.

Table 4.3: Compounds in Milli-Q Water

Compounds	Concentrations (mg/L)
Chloride	0.0
Sulphate	0.0
Cyanide	0.0
Total suspended solids	0.0

4.1.4 Standard Calibration Curve

Every solution used in the experiments was first adjusted to pH 12 before adding any solution that contained cyanide. This was to avoid the free cyanide HCN from vaporising into the air. A standard solution of cyanide was prepared by adding 0.125 g of KCN in 1 L of adjusted pH of Milli-Q water. This gave an approximate 50mg/L of cyanide concentration of the solution. The calculation of KCN weight added in 1 L of water is shown in Appendix C. The amount of standard solution required to prepare synthetic water with a certain concentration of cyanide was based on the calibration curve in Figure 4.1, where y is the cyanide concentration in 1 L of synthetic water and x is the amount of standard solution needed.

The cyanide concentration in synthetic water obtained through this calibration curve showed minor difference because some of the CN^- ions in water were still attached with sodium ions from NaOH that added into the solution earlier.

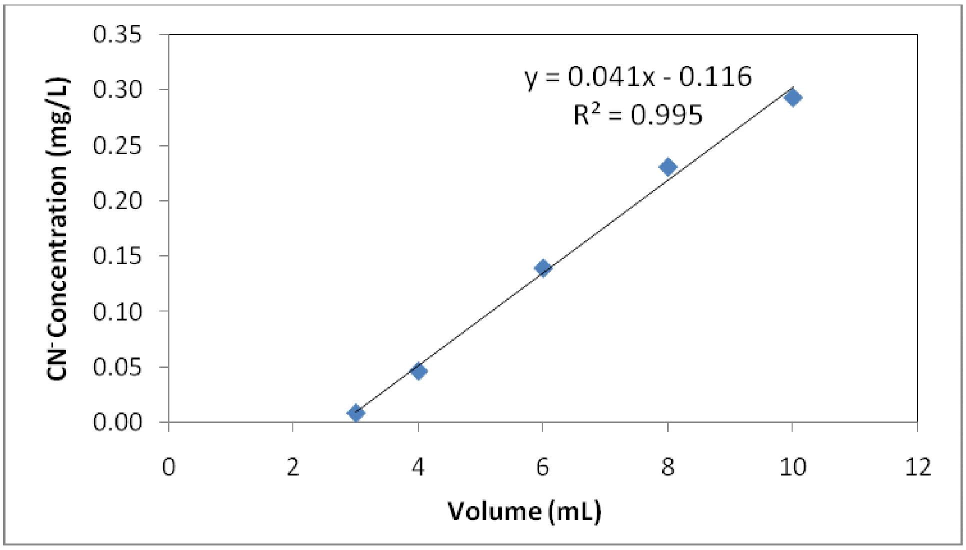


Figure 4.2: Calibration Curve for 1 L Basis

4.2 Effect of Different TiO_2 Concentrations

Firstly, the experiments with different TiO_2 concentrations were performed for six hours and the raw data for this experiment is attached in Appendix D. The removal efficiency, rate constant and *p*-value were calculated. Sample calculations of these figure is shown in Appendix D.

Figure 4.3 shows the removal efficiency based on different TiO_2 concentrations. The removal efficiency of each TiO_2 concentration increased along with time until they reached a certain level (95-100%) and remained constant. In Figure 4.3, the concentration of 1.0 g/L TiO_2 showed the highest removal efficiency among the other concentrations. It was able to remove the cyanide faster than the others and able exceed 90% of removal efficiency after 3 hours of operation time. The second highest removal efficiency was 1.5 g/L TiO_2, whereas the TiO_2 concentrations of 2.0 g/L and 0.5 g/L were almost the same.

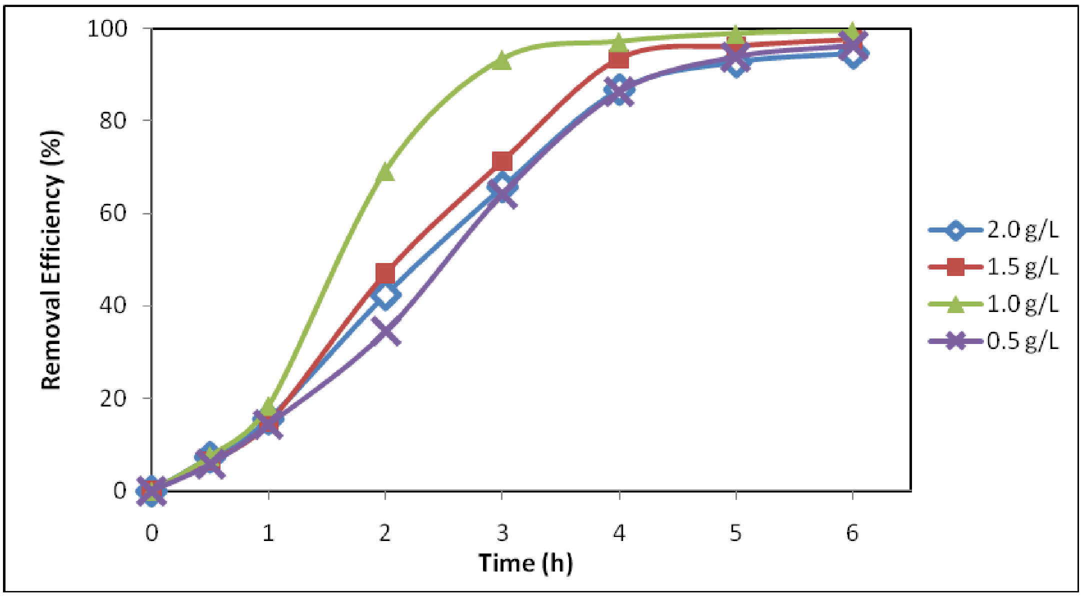
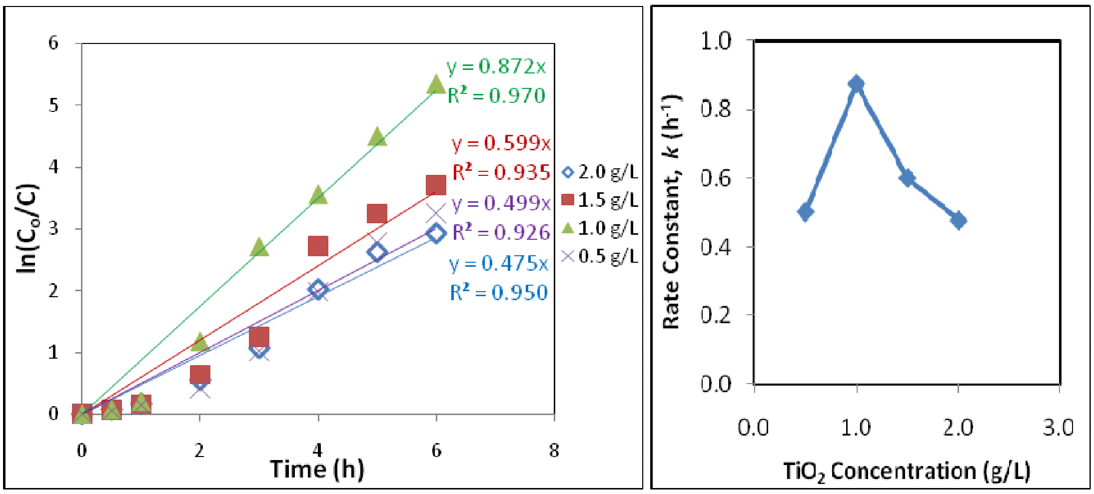


Figure 4.3: Removal Efficiency at Different TiO_2 Concentrations

The experiment data was used to further calculate the $\ln(C_o/C)$ and then plotted a graph as shown in Figure 4.4 (a). Then the First Order Kinetic Model's rate constant, k (as shown in Equation 3.2) was obtained from the slope of the graph. Higher rate constant means greater degradation of cyanide. The relationship between the rate constant and TiO_2 concentration were clearly illustrated in Figure 4.4 (b). It was observed that an increase of TiO_2 concentration from 0.5 to 1.0g/L significantly increased the rate constant. The increment was probably due to the increasing volumetric photon absorption by the TiO_2 (photocatalyst), and therefore providing a higher of the charge carrier per unit volume of cyanide oxidation (Chiang et al., 2003). However, further increase in TiO_2 concentration after the 1.0g/L, the rate constant decreased. This was because the opacity of the suspension increased with the increase of TiO_2 concentration, resulting in that some of the catalyst particles were unable to fully absorb light for activation (Chiang et al., 2003). Thus, the concentration of 1.0 g/L TiO_2 was the optimum catalyst concentration that gave the best performance of the system.

The p-values for each of the TiO_2 concentration in this parameter were summarised in Table 4.4. All of the values were less than the criterion alpha level (p = 0.05), concluding that the regression line for all of the TiO_2 concentrations in this experiment were statistically significant.

In this study, the optimum photocatalyst concentration was found to be 1.0 g/L TiO_2. Thus, it was selected as the amount of photocatalyst to perform the following experiments in order to get the best performance.



(a) (b)

Figure 4.4: First Order Rate Constant, *k* of Different TiO_2 Concentration, at the Function of (a) Time, (b) TiO_2 Concentration

Table 4.4: *p*-values of Different TiO_2 Concentrations

TiO_2 Concentration	*p*-Value
2.0 mg/L	8.07E-06
1.5 mg/L	2.03E-05
1.0 mg/L	1.08E-06
0.5 mg/L	2.39E-05

4.3 Effect of Different UV Light Powers

The experiment data for this section is attached in Appendix E and the removal efficiency, rate constant and *p*-value were calculated. The calculations were similar to those shown in Appendix D.

The removal efficiency on this parameter is shown in Figure 4.5. It is clearly showed that the 16 W of the UV light power achieved the highest removal efficiency at shortest time, followed by the UV light power of 12, 8 and 4 W. For the 0 W of UV light power (i.e., no UV lamp), no energy from UV light source available in the reactor to activate the photocatalysis, and subsequently resulting in no photocatalytic degradation of cyanide during the experiment. As a result, no significant of removal efficiency was observed (Figurer 4.5). However the removal efficiency at 0 Wt condition still showed very minor removal efficiency, which was less than 10% due to several factors. Firstly, the oxygen content in the water could build up a little oxidation reaction of the cyanide and the cyanide concentrations were too low that caused difficulties in measuring it using the spectrophotometer. Besides, the inconsistence of adjusting water sample's pH value also caused the different amount of free cyanide ions released from NaOH alkali, and hence leads to inaccurate cyanide concentration detected in spectrophotometer.

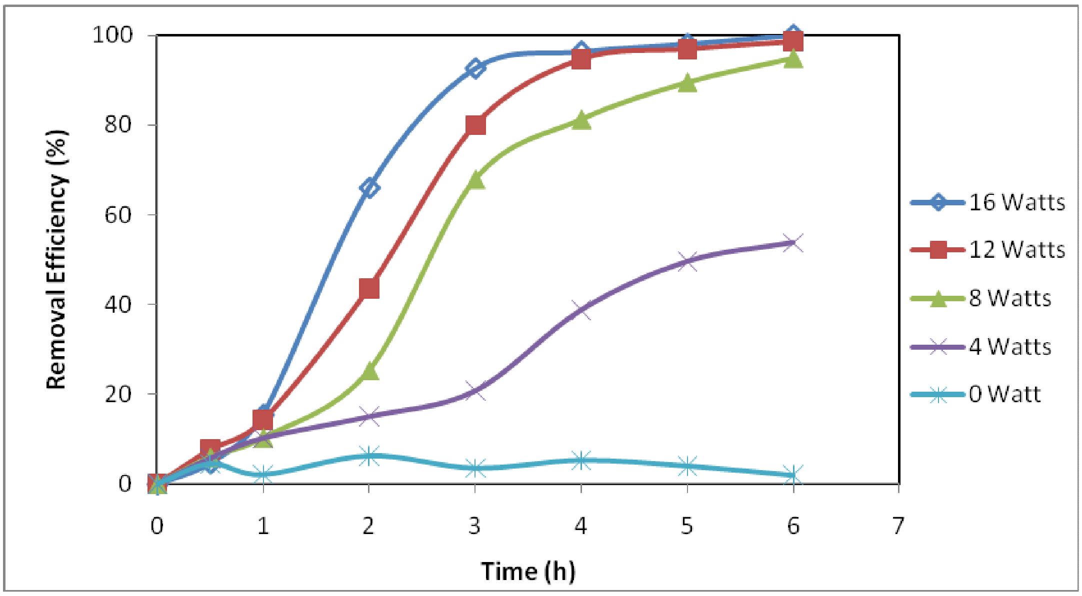
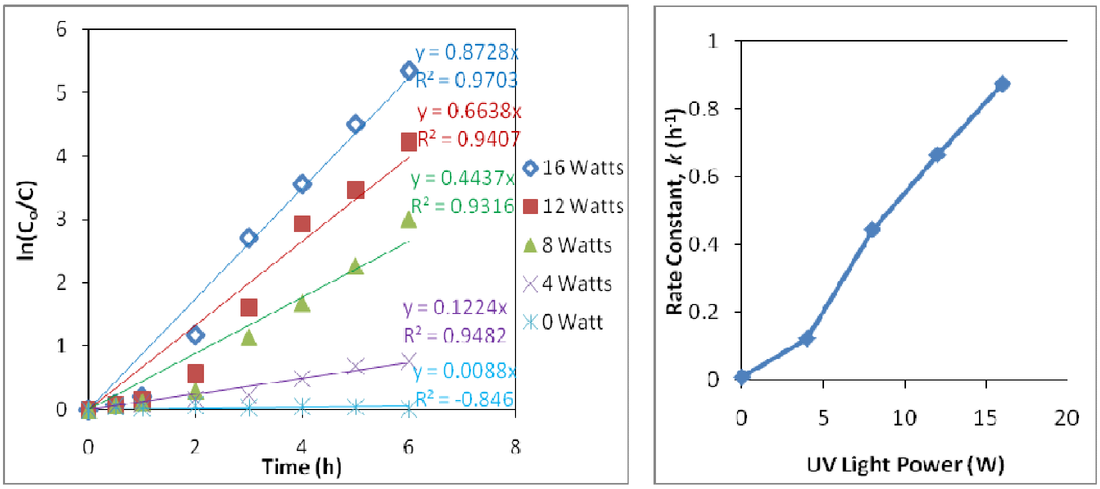


Figure 4.5: Removal Efficiency at Different UV Light Powers

The graph of ln(C_o/C) against time and rate constant against UV light power are shown in Figures 4.6 (a) and (b), respectively. Then, the First Order Kinetic Model's rate constant, *k* for each of the variable was obtained from the slope of the graph in Figure 4.6 (a). It is clearly showed that as the increase of UV light power would cause *k* to increase in the Figure 4.6 (b). This was because the increase of light power in the reactor allows higher rate of electron-hole pairs generated by the photocatalyst and then enhances the degradation rate of the cyanide in the reactor. Thus, higher UV light power provides higher reaction rate and greater *k*.

The *p*-values for each of the light power in this parameter are summarised in Table 4.5. All of the values were less than the criterion alpha level (p = 0.05), except the *p*-value (p = 0.6507) for the experiment without using any UV light (0 W). The *p*-value which was less than the alpha level shows that the regression line of UV light powers of 16 W, 12 W, 8 W and 4 W were statistically significant.

Since the UV light of 16 W provided the best performance in removal efficiency and rate constant compared to the others, it was chosen as the power to perform the following experiments.



(a) **(b)**

Figure 4.6: First Order Rate Constant, *k* at Different Light Power, at the Function of (a) Time, (b) UV Light Power

Table 4.5: ***p*-value of Different UV Light Powers**

Light Power	*P*-Value
16 W	1.08E-06
12 W	1.18E-05
8 W	1.82E-05
4 W	2.56E-05
0 W	0.6507

4.4 Effect of Different Initial Cyanide Concentrations

The experiment data for this section is attached in Appendix F. The removal efficiency, rate constant and *p*-value were calculated, where the calculations were similar to the calculations in Appendix D.

The cyanide concentration profile for this experiment is shown in Figure 4.7, demonstrating that the cyanide concentration in water decreased along the reaction time and became almost constant when the cyanide concentration in water was below 0.01 mg/L. The maximum allowable concentration (MAC) in drinking water set by the Ministry of Health Malaysia is 0.07 mg/L. It is very important to fulfil the regulatory limit in order to ensure this photocatalysis-membrane hybrid system is applicable to the nation. From Figure 4.7, it showed that all of the experiments were able to achieve the regulatory limits of 0.07 mg/L at different initial cyanide concentrations. The lowest initial cyanide concentration (0.91 mg/L) was able to reach the limit within 1 hour of photocatalytic reaction time. However, the highest initial cyanide concentration (0.30 mg/L) reached the limit at around 2.5 hour. Therefore, lower initial cyanide concentration could achieve the limit at shorter time.

The removal efficiency for this parameter is shown in Figure 4.8. It clearly showed that the lowest initial cyanide concentration (0.09 mg/L) had the greatest removal efficiency during the shortest period of time. This was because lower cyanide concentration was more easily to oxidise as there were many positively charged hole generated from the photocatalyst available. On the other hand, the other initial concentrations also depicted almost the same trend in the graph due to higher cyanide concentration requiring more positively charge hole for oxidation. Besides, all of the experiments in this section were able to achieve almost 100% of removal efficiency within the 6 hours period.

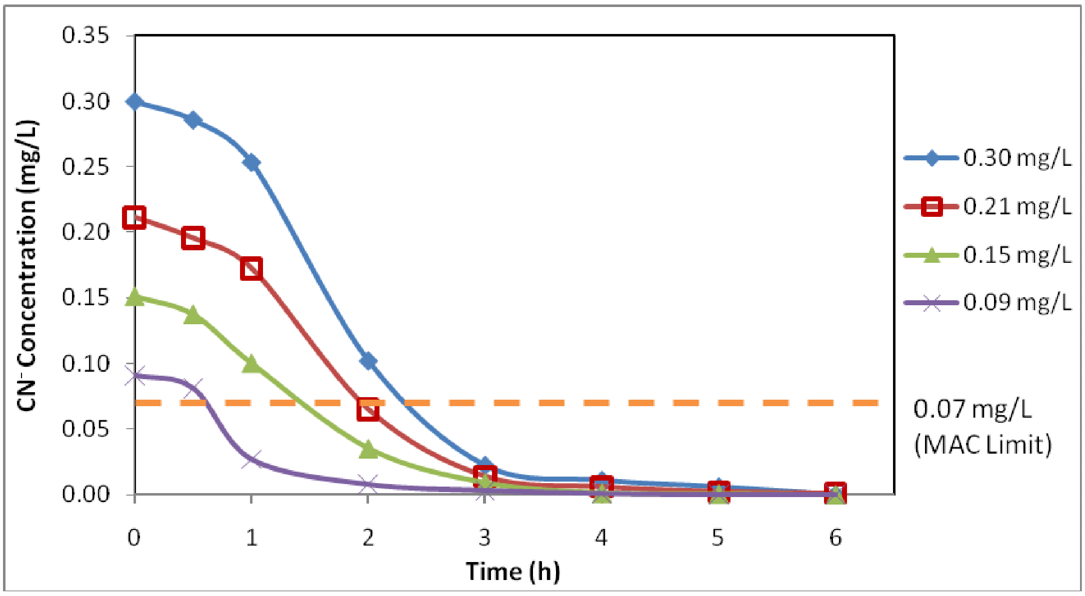


Figure 4.7: Concentration Profile

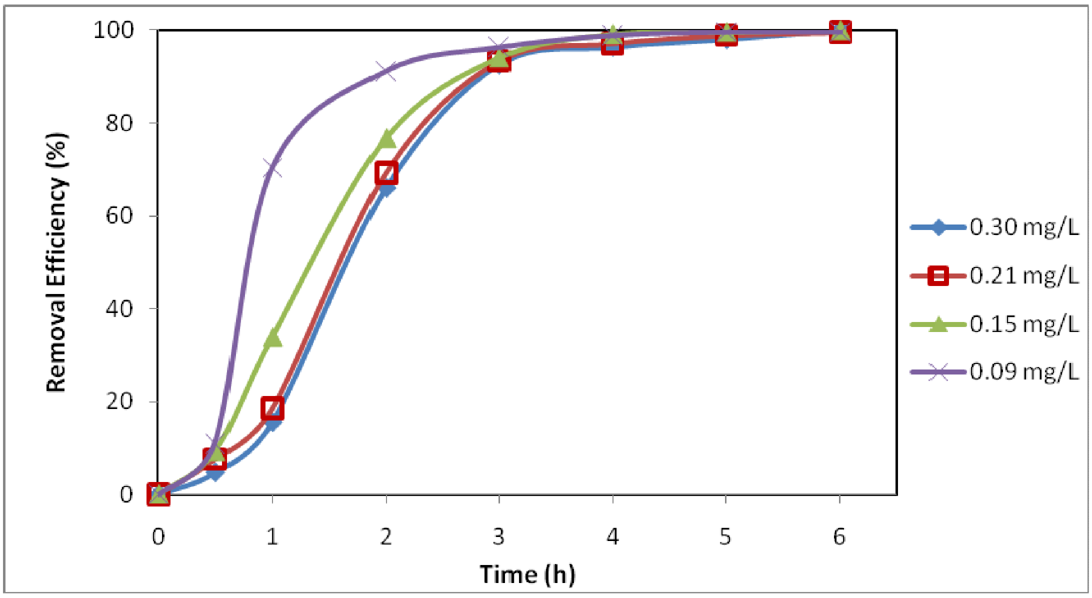
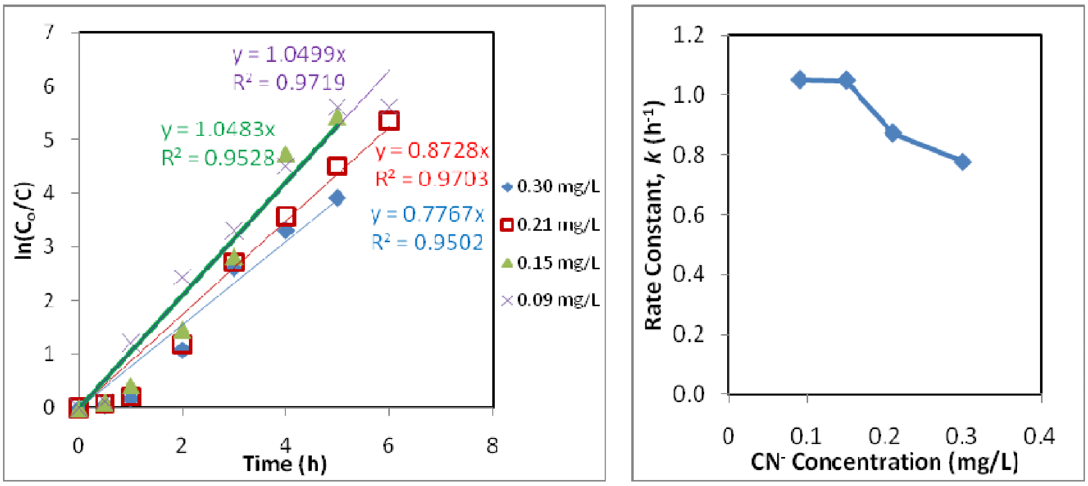


Figure 4.8: Removal Efficiency at Different Initial Cyanide (CN^-) Concentrations

The graph of ln(C_o/C) against time is plotted in Figure 4.9 (a), where the slope of each of the line is their rate constant, *k*. Figure 4.9 (b) shows the rate constant against initial cyanide concentration. Each of the *k* values in this parameter was relatively high (0.7 to 1.1 h^{-1}) because of the optimum TiO_2 concentration (1.0 g/L) and highest UV light power (16 W) are applied into the experiments, and thus providing the best performance and greatest *k* along the experiments. Based on the Figure 4.9 (b), *k* was almost constant when increasing the initial cyanide concentration from 0.09 to 0.15 mg/L, but further increasing caused *k* to decrease. This situation happened because during the low initial cyanide concentration, the electron-hole pairs generated from photocatalyst were more than the cyanide ions available for oxidation. However, further increasing the initial cyanide concentration from 0.15 mg/L resulted in insufficient positively charged hole for oxidation and longer time was required to regenerate the electron-hole pairs, thus the rate constant, *k* was decreased.

The *p*-values for the line regression for each of the initial cyanide concentration in this parameter are summarised in Table 4.6. All of the values were less than the criterion alpha level (p = 0.05), implying that all of the regressions for different initial cyanide concentrations were statistically significant.



(a) **(b)**

Figure 4.9: First Order Rate Constant, *k* at Different Initial Cyanide (CN^-) Concentrations, at the Function of (a) Time, (b) CN^- Concentration

Table 4.6: *p*-value of Different Initial Cyanide (CN^-) Concentrations

CN^- Concentration	*P*-Value
0.30 mg/L	5.35E-05
0.21 mg/L	1.08E-06
0.15 mg/L	3.80E-05
0.09 mg/L	6.69E-06

4.5 Total Recovery of Photocatalyst

The total suspended solid (TSS) concentration of water samples for each of the experiment is summarised in Appendix G and the total recovery of TiO_2 is calculated using Equation 3.6. The sample calculation is shown in the same appendix. It was easyto observe the precense of TiO_2 in water as TiO_2 would cause turbidity upon the addition. Figure 4.10 shows the water samples taken from feed and permeate. The water sample in feed contained very high concentration of TiO_2, which exhibited a heavy white turbidity in Figure 4.10 (a). However, the turbidity in permeate was lower in Figure 4.10 (b), suggesting that the TiO_2 concentration in the water sample was lower.

The results of total TiO_2 recovery are illustrated in Figure 4.11. Based on the result, the total recovery of TiO_2 in Experiment A showed an increasing recovery percentage as the initial TiO_2 concentration decreased. This was because the hollow fiber membrane in the system was unable to filter out large concentration of photocatalyst, and thus leaving a relatively high TSS concentration of TiO_2 in permeate and low total recovery was obtained. In contrast, the lowest initial TiO_2 concentration which was 0.5 g/L demonstrating the highest recovery (96.6 %) since little amount of TiO_2 was easily filtered out by the hollow fiber membrane.

Total TiO_2 recovery in Experiments B and C did not show a clear trend. This was because the same initial amount of TiO_2 concentrations were used in both of the experiments. Therefore, the total recovery for each of the experiment were almost the same, rangingfrom 95.7 to 96.5 %. In order to improve the total recovery of TiO_2 and obtain purer permeate water, a improvement of the membrane is required.

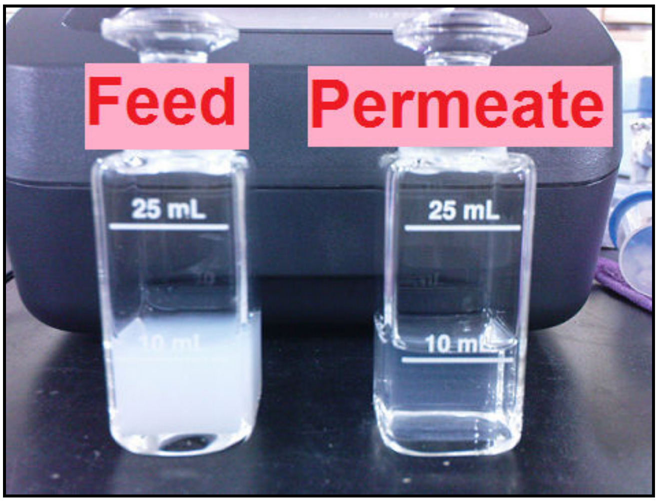


Figure 4.10: Water Samples of TiO_2 Feed Solution (Left) and Permeate Solution (Right)

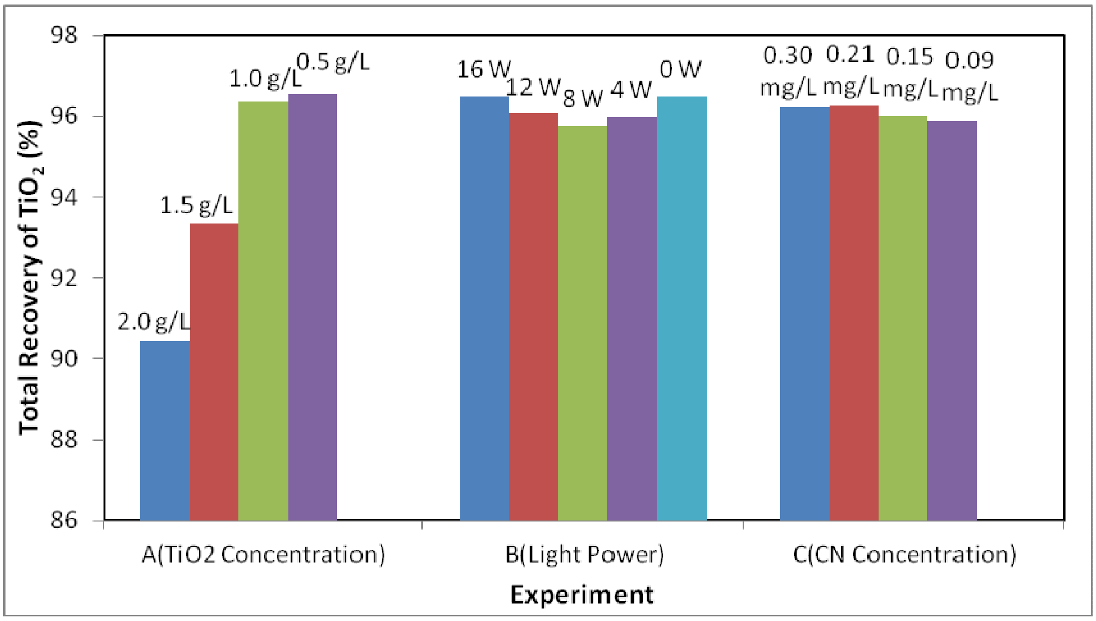


Figure 4.11: Total Recovery of TiO_2 in Different Experiments

4.6 Problems Encountered

In this study, several problems were identified when performing experiments and analysing data. The problems encountered are as followed:

(a) Inconsistent method of adjusting water sample's pH value also caused different amount of free cyanide ion being released from the alkali water, and hence leading to inaccurate cyanide concentration detected in spectrophotometer. Moreover, the excess acid added to the water sample during the adjustment might also cause some of the cyanide ion in water to vaporise, and then decreasing the accuracy of cyanide concentration detected in the water sample.

(b) The detectable range of cyanide concentration by spectrophotometer is narrow (0.002 to 0.240 mg/L), and result in limitation barrier of the study. The initial cyanide concentration used in this study was therefore very low. This could lead to the misleading of the actual trend pattern of the degradation in concentration profile and removal efficiency graphs. Chiang et al. (2003) used a much higher initial cyanide concentration, which is 10 – 100 mg/L of cyanide in water in their study. The authors obtained straight trend lines in concentration profiles which were then able to directly obtain the photocatalytic degradation reaction rate from the slope of the lines.

(c) Aeration system was not applied to the reactor and this caused slow degradation of cyanide in water. Since the reactor was closed when performing the experiment, the air inside the tank was limited. The oxygen level in the reactor decreased along the reaction because the oxidation process occurred in the photocatalysis process. According to Dabrowski et al. (2002), increasing oxygen flow rate to the photocatalysis system was able to increase the photocatalytic oxidation of cyanide. Osathaphan et al. (2008) found out that the optimum aeration rate was 2.2 L/min, and further increasing of the flow rate would cause decrease in cyanide oxidation rate. This was due to bubbles inside the suspension were too big until they hindered the UV light path, and thus affected the overall oxidation process.

Hence, the oxygen concentrations in the reactor can affect the degradation rate of the experiments.

(d) Last but not least, the hollow fiber membrane used in this system did not give highly purified water permeate, where the maximum recovery of TiO_2 was 96.6 % only. This situation happened possibly because of the particle size of TiO_2 used in this study were slightly smaller than the membrane's pore size (not measured in this study). Therefore, TiO_2 particles could be still found in the permeate water.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Based on the experiments performed with different type of initial TiO_2 concentrations, UV light powers and initial cyanide concentrations, the following conclusions were established:

(a) The optimum concentration of TiO_2 to provide the best performance of the cyanide degradation was 1 g/L, as it provided the highest removal efficiency in shorter time and also gave the highest first order rate constant. The TiO_2 concentration lower than this optimum value was not able to provide sufficient charge carrier per unit volume for cyanide oxidation, decreased the removal efficiency and rate constant. However, a higher TiO_2 concentration than the optimum value had caused UV light unable to reach every TiO_2 to activate them, decreased the rate of degradation.

(b) The optimum power of UV light found in this study was 16 W because it achieved the highest removal efficiency and first order rate constant among the other powers. The increase of UV light power in the reactor increased the amount of electron-hole pairs generated from photocatalyst, and thus increased the removal efficiency and rate constant.

(c) Initial cyanide concentration would affect the removal efficiency and rate constant of the cyanide degradation. The higher the initial cyanide concentration, the longer time it required to degrade in order to reach the MOC limit. Besides, higher initial concentration also led to lower removal efficiency and rate constant.

(d) The total recovery of TiO_2 was important in this study to show the purity level of water in permeates. It was strongly affected by the TiO_2 concentration used in feed, where high TiO_2 concentration in feed could bring failure to the membrane, and thus reduce the percentage of total recovery of TiO_2.

In summary, this photocatalysis-membrane hybrid system was applicable to the industries to improve water quality. Because this system has the advantages of using inexpensive materials and ease of set up, it is suitable to use at home, or even at remote place where the water treatment plant is not available at that area.

5.2 Recommendations

This study involved improving the water quality, which is very useful in water treatment development. During this study, some problems were encountered and can be improved. Some recommendations are proposed in this section and further investigations on the improvement are needed.

Firstly, the design of the system should include an aeration system inside the reactor. This is to provide a better air flow and enhance the oxidation reactions. However, the aeration rate should not be too high because of excessive bubbles in the water can hinder the path of UV light and then affected the oxidation process.

Other than that, the hollow fiber membrane with known size should be used to improve the removal of photocatalyst from water. Further study on using the membrane which traps photocatalyst inside can be carried out. This type of design allows the reuse of photocatalst and might provide better filtration.

Next, the effect of pH on removal efficiency of cyanide in water can be used as one of the parameter for future study. Since the pH of water can affect the degree of cyanide dissolved in water, it might be a great influence factor in the removal efficiency and rate constant of the study.

The current method to analyse cyanide concentration present in water sample, which is spectrophotometer, is not that suitable for analysing the cyanide concentration in this study. Hence, further study should replace it with Ion Chromatography because it is able to measure wide range of concentrations of major anions and cations (Bruckner, 2010). This instrument is widely used in water chemistry analysis; and hence it might be a good choice to use in this study.

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APPENDICES

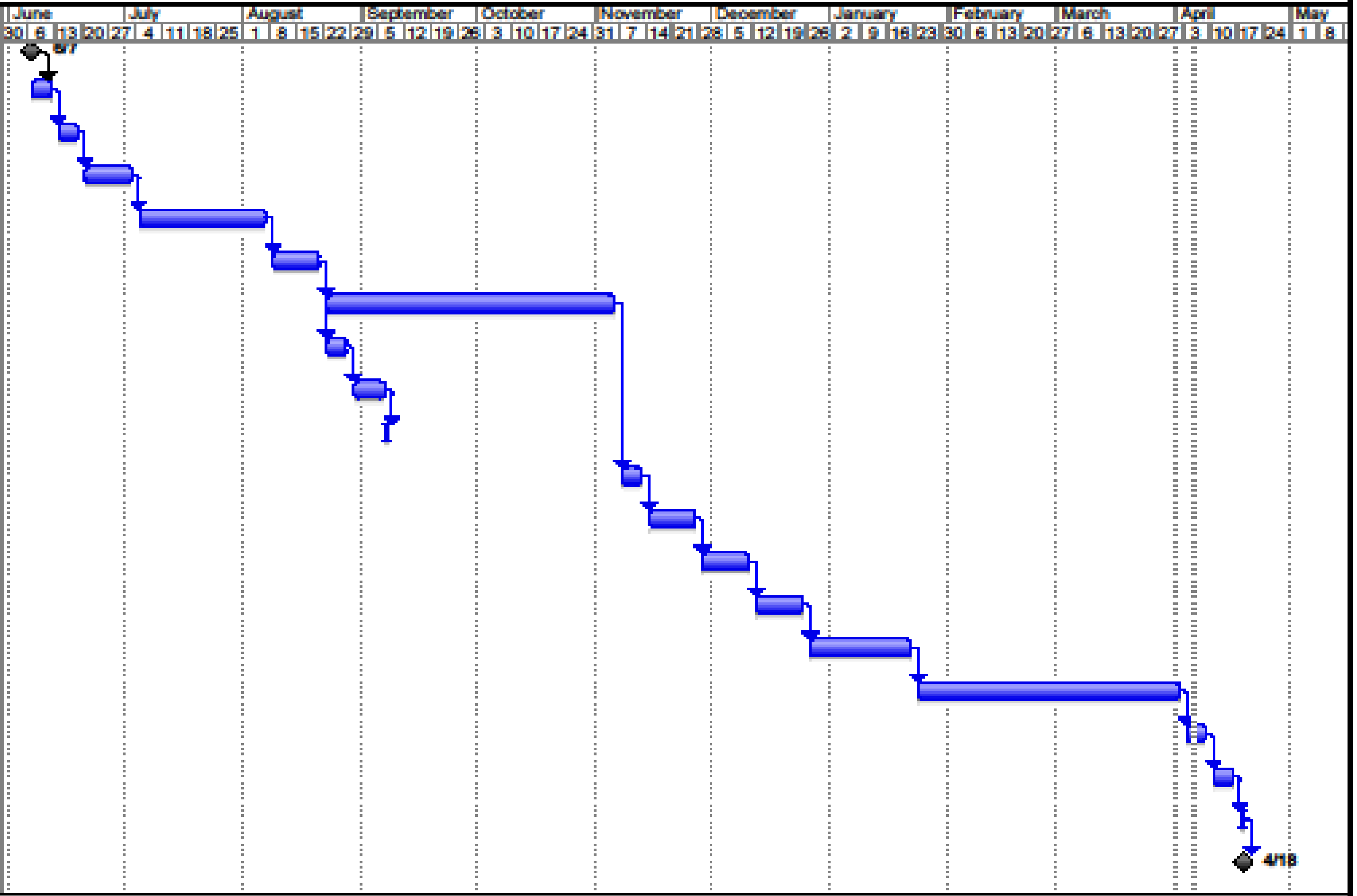
APPENDIX A: Gantt Chart

The schedule of this project was done by using Microsoft Office Project. The Gantt chart of this project is attached on the following page.

Universiti Tunku Abdul Rahman

Faculty of Engineering & Science
Department of Chemical Engineering
Final Year Project
Master Schedule

ID		Task Name	% Complete
1	✓	Start	100%
2	✓	Understanding Title	100%
3	✓	Research on Related Information	100%
4	✓	Introduction	100%
5	✓	Literature Review	100%
6	✓	Methodology	100%
7	✓	Purchasing	100%
8	✓	Progress Report Preparation	100%
9	✓	Presentation I Preparation	100%
10	✓	Presentation I	100%
11	✓	Apparatus Set Up	100%
12	✓	Experiment A	100%
13	✓	Experiment B	100%
14	✓	Experiment C	100%
15	✓	Data Analysis	100%
16	✓	Result & Discussion	100%
17	✓	Conclusion & Recommendation	100%
18	✓	Final Report Preparation	100%
19	✓	Presentation II	100%
20	✓	Finish	100%



Project: Project Schedule
Date: Wed 4/6/11

Task	☐	Progress	▬	Summary	▬	External Tasks	▬	Deadline	⇩
Split	....	Milestone	◆	Project Summary	▬	External Milestone	◆		

Title: Removal of Cyanide Using Photocatalysis Membrane
Hybrid System

Reported by:
Liau Kee Fui

Committed Signature

Date: 6th April 2011

APPENDIX B: Material Safety Data Sheet (MSDS)

Chemical Safety Data: Potassium Cyanide

Common synonyms	-
Formula	KCN
Physical properties	Form: white crystalline solid or powder Stability: Stable, but sensitive both to light and to moisture Melting point: 634ºC Specific gravity: 1.52
Principal hazards	• Like most cyanides, potassium cyanide is highly toxic and may be fatal if swallowed. If can also cause serious harm, or be fatal, if inhaled or absorbed through the skin. • Potassium cyanide reacts readily with acids to generate hydrogen cyanide gas (HCN) which is extremely toxic. • Skin contact may lead to burns.
Safe handling	Wear safety glasses and gloves. Work in a well ventilated area, preferably using a fume hood. Take advice from a trained chemist before starting work with cyanides. If cyanides are to be used more than very occasionally, ensure that a cyanide antidote kit is available at all times.
Emergency	• Eye contact: Immediately flush the eye with water. Call for medical help. • Skin contact: Immediately wash off with soap and water. Call for medical help if the skin appears red or blistered. • If swallowed: Call for immediate medical help - ingestion of cyanide is a medical emergency. Use the cyanide antidote kit if one is available.
Disposal	Store for later disposal as hazardous solid waste or for chemical destruction.
Protective equipment	Safety glasses, gloves.

Source: http://cartwright.chem.ox.ac.uk/hsci/chemicals/potassium_cyanide.html,
April 18, 2005.

Chemical Safety Data: Sodium Hydroxide

Common synonyms	Caustic soda, soda lye
Formula	NaOH
Physical properties	Form: White semi-transparent solid, often supplied as pellets weighing about 0.1g Stability: Stable, but hygroscopic. Absorbs carbon dioxide from the air. Melting point: 318ºC Water solubility: high (dissolution is very exothermic) Specific gravity: 2.12
Principal hazards	• Contact with the eyes can cause serious long-term damage • The solid and its solutions are corrosive • Significant heat is released when sodium hydroxide dissolves in water
Safe handling	Always wear safety glasses. Do not allow solid or solution to come into contact with your skin. When preparing solutions swirl the liquid constantly to prevent "hot spots" developing.
Emergency	• Eye contact: Immediately flush the eye with plenty of water. Continue for at least ten minutes and call for immediate medical help. • Skin contact: Wash off with plenty of water. Remove any contaminated clothing. If the skin reddens or appears damaged, call for medical aid. • If swallowed: If the patient is conscious, wash out the mouth well with water. Do not try to induce vomitting. Call for immediate medical help
Disposal	Small amounts of dilute sodium hydroxide can be flushed down a sink with a large quantity of water, unless local rules prohibit this. Larger amounts should be neutralised before disposal.
Protective equipment	Always wear safety glasses when handling sodium hydroxide or its solutions. If you need gloves, neoprene, nitrile or natural rubber are suitable for handling solutions at concentrations of up to 70%.

Source: http://cartwright.chem.ox.ac.uk/hsci/chemicals/sodium_hydroxide.html, August 31, 2006.

APPENDIX C: Preparation

Table C.1: Circumference and Diameter of Reactor

Readings	**Reactor**		**Working Volume**	
	Circumference, C_T (cm)	Diameter, $D_T\,(\text{cm}) = \frac{C_T}{\pi}$	Circumference, C_W (cm)	Diameter, $D_W\,(\text{cm}) = \frac{C_W}{\pi}$
1	50.0	15.92	50.0	15.92
2	52.1	16.58	51.6	16.42
3	53.2	16.93	52.2	16.62
4	56.1	17.86	53.2	16.93
5	57.6	18.33	54.0	17.19
Average	53.8	17.13	52.2	16.62

Reactor Tank

Tank Height, H_T = 26.1 cm = 0.261 m

Tank Volume, $V_T = \frac{\pi}{4} \times D_T{}^2 \times H_T$

$$= \frac{\pi}{4} \times 17.13^2 \times 26.1$$

$$= 6011.7\ \text{cm}^3$$

$$= 6011.7\ \text{mL}$$

$$= 6.01\ \text{L}$$

Working Volume

Solution Volume, V_W = 4.00 L

Water Level in Tank, H_W = 20 cm = 0.200 m

1. Cyanide Solution

Molecular weight of potassium cyanide (KCN) = 65.12 g/mol

Molecular weight of cyanide (CN^-) = 26.02 g/mol

Water volume = 1 L

$$\text{Mass of KCN} = 0.050\ \text{g/L} \times \frac{65.12\ \text{g/mol}}{26.02\ \text{g/mol}} \times 1\ \text{L} = 0.125\ \text{g}$$

Hence, the mass of KCN required to dissolve in 1 L water to produce a cyanide standard solution with 50 mg/L CN^- is **0.125 g**.

Table C.2: Concentration of Water Sample after Dilution

Volume of Standard Solution (mL)	Dilution Volume (mL)	Concentration (mg/L CN^-)			
		1	2	3	Average
3	1000	0.008	0.008	0.008	0.008
4	1000	0.046	0.046	0.046	0.046
6	1000	0.139	0.139	0.139	0.139
8	1000	0.230	0.231	0.230	0.230
10	1000	0.292	0.293	0.294	0.293

From the calibration graph in Figure 4.1, y = 0.0419x – 0.1163

where y = cyanide concentration

x = volume of cyanide standard solution required for 1 L

Sample Calculation

To prepare 4 L of 0.2 mg/L cyanide:

$0.2 = 0.0419x - 0.1163$

$x = 7.5489$ mL

Total volume of cyanide standard solution required = 4 × 7.5489 = **30.2 mL**

APPENDIX D: Effect of Different TiO_2 Concentrations

Table D.1: Experimental Data on Different TiO_2 Concentrations

Initial TiO_2 Concentration (g/L)	Time (h)	Cyanide Concentration, *C* (mg/L)				Removal Efficiency (%)	ln(C_0/C)	TSS (mg/L)
		1	2	3	Average			
2.0	0	0.194	0.195	0.195	0.195	0.00	0.000	2
	0.5	0.180	0.180	0.181	0.180	7.36	0.076	152
	1	0.165	0.164	0.164	0.164	15.58	0.169	186
	2	0.112	0.112	0.112	0.112	42.47	0.553	214
	3	0.066	0.067	0.067	0.067	65.75	1.072	247
	4	0.025	0.026	0.026	0.026	86.82	2.026	270
	5	0.014	0.014	0.014	0.014	92.81	2.632	201
	6	0.011	0.010	0.010	0.010	94.69	2.936	141
							Average	177
1.5	0	0.207	0.208	0.208	0.208	0.00	0.000	0
	0.5	0.195	0.194	0.194	0.194	6.42	0.066	53
	1	0.177	0.177	0.177	0.177	14.77	0.16	65
	2	0.110	0.110	0.110	0.110	47.03	0.635	69
	3	0.076	0.076	0.027	0.060	71.27	1.247	70
	4	0.013	0.014	0.014	0.014	93.42	2.721	70
	5	0.008	0.008	0.008	0.008	96.15	3.256	70
	6	0.005	0.005	0.005	0.005	97.59	3.726	70
							Average	58
1.0	0	0.211	0.212	0.211	0.211	0.00	0.000	2
	0.5	0.195	0.195	0.196	0.195	7.57	0.079	39
	1	0.172	0.173	0.172	0.172	18.45	0.204	43
	2	0.065	0.065	0.065	0.065	69.24	1.179	59
	3	0.014	0.014	0.014	0.014	93.38	2.714	30
	4	0.006	0.006	0.006	0.006	97.16	3.562	61
	5	0.002	0.002	0.003	0.002	98.9	4.506	50
	6	0.001	0.001	0.001	0.001	99.53	5.353	49
							Average	42

0.5	0	0.211	0.211	0.211	0.211	0.00	0.000	0
	0.5	0.199	0.198	0.199	0.199	5.85	0.06	19
	1	0.181	0.181	0.18	0.181	14.38	0.155	22
	2	0.139	0.138	0.138	0.138	34.44	0.422	25
	3	0.075	0.075	0.077	0.076	64.14	1.026	18
	4	0.029	0.029	0.029	0.029	86.26	1.985	21
	5	0.013	0.013	0.013	0.013	93.84	2.787	26
	6	0.008	0.008	0.008	0.008	96.21	3.272	33
							Average	21

Sample calculation for 2.0 g/L initial TiO_2

From Table D.1,

- Initial cyanide concentration = 0.195 mg/L
- Time = 3 hour
- Average concentration of cyanide in water sample = 0.067 mg/L

From Equation 3.1,

Removal Efficiency, $\eta = \left(1 - \frac{C}{C_o}\right) \times 100\%$

$$\eta = \left(1 - \frac{0.067}{0.195}\right) \times 100\%$$

$$\eta = \mathbf{65.75\ \%}$$

To obtain First Order Kinetic rate constant, *k*, a graph of $\ln\left(\frac{C_o}{C}\right)$ against time is needed to plot, where the slope of the graph is the rate constant, *k*. The line equation is followed the Equation 3.5, which is $\ln\left(\frac{C_o}{C}\right) = kt$

$$\ln\left(\frac{C_o}{C}\right) = \ln\left(\frac{0.195}{0.067}\right)$$

$$\ln\left(\frac{C_o}{C}\right) = \mathbf{1.072}$$

Table D.2: Summary Output of Regression Data Imported from Excel
(Different TiO_2 concentrations)

For TiO_2 concentration = 2.0 g/L

Regression Statistics	
Multiple R	0.9852
R Square	0.9706
Adjusted R Square	0.9656
Standard Error	0.4053
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.4833	32.4833	197.7830	8.07E-06
Residual	6	0.9854	0.1642		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.5493	0.2089	2.6294	0.0391	0.0381	1.0605	0.0381	1.0605
2.0 g/L	1.8073	0.1285	14.063	8.07E-06	1.4929	2.1218	1.4929	2.1218

For TiO_2 concentration = 1.5 g/L

Regression Statistics	
Multiple R	0.9798
R Square	0.9600
Adjusted R Square	0.9533
Standard Error	0.4725
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.1293	32.1293	143.9237	2.03E-05
Residual	6	1.3394	0.2232		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.6234	0.2398	2.599	0.0407	0.0366	1.2102	0.0366	1.2102
1.5 g/L	1.3979	0.1165	11.997	2.03E-05	1.1128	1.6830	1.1128	1.6830

For TiO_2 concentration = 1.0 g/L

Regression Statistics	
Multiple R	0.9924
R Square	0.9849
Adjusted R Square	0.9824
Standard Error	0.2903
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.9633	32.9633	391.2634	1.08E-06
Residual	6	0.5055	0.0842		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.4461	0.1529	2.9180	0.0267	0.0720	0.8202	0.0720	0.8202
1.0 g/L	1.0190	0.0515	19.780	1.08E-06	0.8929	1.1450	0.8929	1.1450

For TiO_2 concentration = 0.5 g/L

Regression Statistics	
Multiple R	0.9787
R Square	0.9578
Adjusted R Square	0.9508
Standard Error	0.4852
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.0565	32.0565	136.1945	2.39E-05
Residual	6	1.4122	0.2354		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.6954	0.2420	2.8739	0.0283	0.1033	1.2876	0.1033	1.2876
0.5 g/L	1.6417	0.1407	11.670	2.39E-05	1.2975	1.9860	1.2975	1.9860

APPENDIX E: Effect of Different UV Light Powers

Table E.1: Experimental Data on Different UV Light Powers

Light Power (W)	Time (h)	Cyanide Concentration, *C* (mg/L)				Removal Efficiency (%)	$\ln(C_0/C)$	TSS (mg/L)
		1	2	3	Average			
16	0	0.211	0.212	0.211	0.211	0.00	0.000	2
	0.5	0.195	0.195	0.196	0.195	7.57	0.079	39
	1	0.172	0.173	0.172	0.172	18.45	0.204	43
	2	0.065	0.065	0.065	0.065	69.24	1.179	59
	3	0.014	0.014	0.014	0.014	93.38	2.714	30
	4	0.006	0.006	0.006	0.006	97.16	3.562	61
	5	0.002	0.002	0.003	0.002	98.90	4.506	50
	6	0.001	0.001	0.001	0.001	99.53	5.353	49
							Average	42
12	0	0.204	0.205	0.205	0.205	0.00	0.000	1
	0.5	0.189	0.189	0.189	0.189	7.65	0.080	25
	1	0.175	0.176	0.176	0.176	14.17	0.153	40
	2	0.115	0.115	0.116	0.115	43.65	0.574	64
	3	0.041	0.041	0.041	0.041	79.97	1.608	64
	4	0.011	0.011	0.011	0.011	94.63	2.923	62
	5	0.007	0.006	0.006	0.006	96.91	3.476	53
	6	0.003	0.003	0.003	0.003	98.53	4.223	54
							Average	45
8	0	0.224	0.225	0.225	0.225	0.00	0.000	1
	0.5	0.208	0.209	0.209	0.209	6.01	0.074	36
	1	0.199	0.199	0.199	0.199	10.36	0.121	52
	2	0.165	0.166	0.166	0.166	25.38	0.305	59
	3	0.071	0.071	0.071	0.071	68.02	1.152	61
	4	0.042	0.041	0.041	0.041	81.38	1.693	58
	5	0.023	0.023	0.023	0.023	89.64	2.279	57
	6	0.012	0.011	0.010	0.011	95.05	3.017	59
							Average	48

4	0	0.222	0.222	0.222	0.222	0.00	0.000	8
	0.5	0.210	0.209	0.209	0.209	5.71	0.059	56
	1	0.199	0.199	0.200	0.199	10.21	0.108	57
	2	0.189	0.188	0.189	0.189	15.02	0.163	52
	3	0.176	0.176	0.176	0.176	20.72	0.232	50
	4	0.136	0.136	0.136	0.136	38.74	0.490	50
	5	0.112	0.112	0.112	0.112	49.55	0.684	39
	6	0.102	0.103	0.103	0.103	53.75	0.771	40
							Average	44
0	0	0.211	0.211	0.211	0.211	0.00	0.000	1
	0.5	0.201	0.202	0.202	0.202	4.42	0.045	49
	1	0.206	0.207	0.207	0.207	2.05	0.021	39
	2	0.198	0.198	0.198	0.198	6.16	0.064	40
	3	0.203	0.204	0.204	0.204	3.48	0.035	58
	4	0.200	0.200	0.200	0.200	5.21	0.054	45
	5	0.200	0.204	0.204	0.203	3.95	0.040	48
	6	0.207	0.207	0.207	0.207	1.90	0.019	48
							Average	41

Table E.2: Summary Output of Regression Data Imported from Excel (Different UV Light Powers)

For UV light power = 16 W

Regression Statistics	
Multiple R	0.9924
R Square	0.9849
Adjusted R Square	0.9824
Standard Error	0.2903
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.9633	32.9633	391.2634	1.08E-06
Residual	6	0.5055	0.0842		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.4461	0.1529	2.9180	0.0267	0.0720	0.8202	0.0720	0.8202
16 watts	1.0190	0.0515	19.780	1.08E-06	0.8929	1.1450	0.8929	1.1450

For UV light power = 12 W

Regression Statistics	
Multiple R	0.9831
R Square	0.9666
Adjusted R Square	0.9610
Standard Error	0.4318
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.3500	32.3500	173.5030	1.18E-05
Residual	6	1.1187	0.1865		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.6234	0.2188	2.8497	0.0292	0.0881	1.1587	0.0881	1.1587
12 watts	1.2667	0.0962	13.1721	1.18E-05	1.0314	1.5020	1.0314	1.5020

For UV light power = 8 W

Regression Statistics	
Multiple R	0.9805
R Square	0.9614
Adjusted R Square	0.9550
Standard Error	0.4640
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.1772	32.1772	149.4838	1.82E-05
Residual	6	1.2915	0.2153		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.6751	0.2324	2.9051	0.0272	0.1065	1.2437	0.1065	1.2437
8 watts	1.8632	0.1524	12.226	1.82E-05	1.4903	2.2361	1.4903	2.2361

For UV light power = 4 W

Regression Statistics	
Multiple R	0.9782
R Square	0.9568
Adjusted R Square	0.9496
Standard Error	0.4909
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.0230	32.0230	132.9023	2.56E-05
Residual	6	1.4457	0.2410		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.423	0.262	1.612	0.158	-0.219	1.064	-0.219	1.064
4 watts	7.2281	0.6270	11.5283	2.56E-05	5.6939	8.7623	5.6939	8.7623

For UV light power = 0 W

Regression Statistics	
Multiple R	0.1909
R Square	0.0364
Adjusted R Square	-0.1242
Standard Error	2.3184
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	1.2191	1.2191	0.2268	0.6507
Residual	6	32.2497	5.3749		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	1.9840	1.6893	1.1745	0.2847	-2.1495	6.1176	-2.1495	6.1176
0 watt	20.249	42.517	0.4762	0.6507	-83.787	124.284	-83.787	124.284

APPENDIX F: Effect of Different Initial Cyanide Concentrations

Table F.1: Experimental Data on Different Initial Cyanide Concentrations

Initial CN Concentration (mg/L)	Time (h)	Cyanide Concentration, *C* (mg/L)				Removal Efficiency (%)	$\ln(C_0/C)$	TSS (mg/L)
		1	2	3	Average			
0.30	0	0.291	0.310	0.298	0.300	0.00	0.000	8
	0.5	0.285	0.286	0.286	0.286	4.67	0.048	36
	1	0.253	0.253	0.254	0.253	15.46	0.168	40
	2	0.102	0.102	0.102	0.102	65.96	1.078	52
	3	0.022	0.022	0.023	0.022	92.55	2.597	50
	4	0.011	0.011	0.011	0.011	96.33	3.305	50
	5	0.006	0.006	0.006	0.006	98.00	3.911	56
	6	0.000	0.000	0.000	0.000	100.00	-	48
							Average	43
0.21	0	0.211	0.212	0.211	0.211	0.00	0.000	2
	0.5	0.195	0.195	0.196	0.195	7.57	0.079	39
	1	0.172	0.173	0.172	0.172	18.45	0.204	43
	2	0.065	0.065	0.065	0.065	69.24	1.179	59
	3	0.014	0.014	0.014	0.014	93.38	2.714	30
	4	0.006	0.006	0.006	0.006	97.16	3.562	61
	5	0.002	0.002	0.003	0.002	98.90	4.506	50
	6	0.001	0.001	0.001	0.001	99.53	5.353	49
							Average	42
0.15	0	0.151	0.152	0.152	0.152	0.00	0.000	1
	0.5	0.140	0.140	0.133	0.138	9.23	0.097	40
	1	0.101	0.101	0.099	0.100	33.85	0.413	52
	2	0.035	0.036	0.035	0.035	76.70	1.457	56
	3	0.009	0.009	0.009	0.009	94.07	2.824	59
	4	0.002	0.001	0.001	0.001	99.12	4.734	50
	5	0.001	0.001	0.000	0.001	99.56	5.427	56
	6	0.000	0.000	0.000	0.000	100.00	-	48
							Average	45

0.09	0	0.090	0.091	0.092	0.091	0.00	0.000	2
	0.5	0.079	0.082	0.082	0.081	10.99	0.116	37
	1	0.027	0.027	0.027	0.027	70.33	1.215	49
	2	0.008	0.008	0.008	0.008	91.21	2.431	59
	3	0.003	0.004	0.003	0.003	96.34	3.307	58
	4	0.001	0.001	0.001	0.001	98.90	4.511	60
	5	0.001	0.000	0.000	0.000	99.63	5.609	58
	6	0.000	0.001	0.000	0.000	99.63	5.609	40
							Average	45

Table F.2: Summary Output of Regression Data Imported from Excel (Different Cyanide Concentrations)

For Initial Cyanide Concentration = 0.30 mg/L

Regression Statistics	
Multiple R	0.9849
R Square	0.9700
Adjusted R Square	0.9640
Standard Error	0.3543
Observations	7

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	20.3009	20.3009	161.7095	5.35E-05
Residual	5	0.6277	0.1255		
Total	6	20.9286			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.4561	0.1925	2.3697	0.0640	-0.0387	0.9509	-0.0387	0.9509
0.30 mg/L	1.1082	0.0871	12.717	5.35E-05	0.8842	1.3322	0.8842	1.3322

For Initial Cyanide Concentration = 0.21 mg/L

Regression Statistics	
Multiple R	0.9924
R Square	0.9849
Adjusted R Square	0.9824
Standard Error	0.2903
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.9633	32.9633	391.2634	1.08E-06
Residual	6	0.5055	0.0842		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.4461	0.1529	2.9180	0.0267	0.0720	0.8202	0.0720	0.8202
0.21 mg/L	1.0190	0.0515	19.7804	1.08E-06	0.8929	1.1450	0.8929	1.1450

For Initial Cyanide Concentration = 0.15 mg/L

Regression Statistics	
Multiple R	0.9868
R Square	0.9738
Adjusted R Square	0.9686
Standard Error	0.3310
Observations	7

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	20.3807	20.3807	185.9931	3.8E-05
Residual	5	0.5479	0.1096		
Total	6	20.9286			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.4597	0.1795	2.5613	0.0506	-0.0017	0.9210	-0.0017	0.9210
0.15 mg/L	0.8214	0.0602	13.6379	3.8E-05	0.6666	0.9763	0.6666	0.9763

For Initial Cyanide Concentration = 0.09 mg/L

Regression Statistics	
Multiple R	0.9861
R Square	0.9723
Adjusted R Square	0.9677
Standard Error	0.3929
Observations	8

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	32.5427	32.5427	210.840	6.69E-06
Residual	6	0.9261	0.1543		
Total	7	33.4688			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	0.0016	0.2313	0.0070	0.9946	-0.5644	0.5676	-0.5644	0.5676
0.09 mg/L	0.9424	0.0649	14.5203	6.69E-06	0.7836	1.1012	0.7836	1.1012

APPENDIX G: Total Recovery of Photocatalyst

Table G.2: Summary of Total Recovery of TiO_2 from Previous Experiments

Experiment		Total Suspended Solid (mg/L)		Total Recovery of TiO_2, R (%)
		Feed	Average Permeate	
A (TiO_2 Conc.)	2.0 g/L	1853	177	90.45
	1.5 g/L	1487	99	93.34
	1.0 g/L	1150	42	96.35
	0.5 g/L	610	21	96.56
B (Light Power)	16 W	1194	42	96.48
	12 W	1143	45	96.06
	8 W	1129	48	95.75
	4 W	1095	44	95.98
	0 W	1171	41	96.50
C (CN^- Conc.)	0.30 mg/L	1137	43	96.22
	0.21 mg/L	1126	42	96.27
	0.15 mg/L	1124	45	96.00
	0.09 mg/L	1097	45	95.90

Sample Calculation

For Experiment B with light power of 16 W,

TSS in feed, C_F = 1194 mg/L

TSS in permeate, C_P = 42 mg/L

From Equation 3.6,

Percentage Recovery of TiO_2, $R = \left(1 - \frac{C_P}{C_F}\right) \times 100\%$

$$R = \left(1 - \frac{42}{1194}\right) \times 100\%$$

$$R = \mathbf{96.48\ \%}$$